

**International Symposium  
on Epi-Graphene**

**ISEG  
2019**



**TU Chemnitz**  
**25.-28. August 2019**



TECHNISCHE UNIVERSITÄT  
CHEMNITZ

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## Coffee breaks & meals

- There is one coffee break in the morning (10:15) and one in the afternoon (15:15).
- Lunch is served every day 12:30 – 13:30 in the Mensa near the institute.

## Internet access

The most convenient way to access the internet is by using the “eduroam” network. If you are not able to use the “eduroam” network, please use the WLAN “tu-chemnitz.de” and the password:

*iseg-tuc*

When you use the network the first time, you will be forwarded to the login page automatically or you can manually do this <https://mytuc.org/anab> by choosing the menu “Veranstaltung”.

## Excursion

On Wednesday afternoon we will visit the falconry in castle Wolkenstein, an idyllic historical site located 30 km away from Chemnitz in the beautiful Ore Mountains. We will reach this destination by bus leaving at around 14:00. Make sure not to miss it!

## Conference dinner

The conference dinner will take place in the restaurant “Zum Grenadier” in castle Wolkenstein, starting at around 17:00. We will have a variety of local specialties from Saxony. The bus will take us back after the dinner at approximately 21:00.



Castle Wolkenstein in the Ore Mountains. © CC BY-SA 3.0 by [Botulph](#).

## Venue of the conference

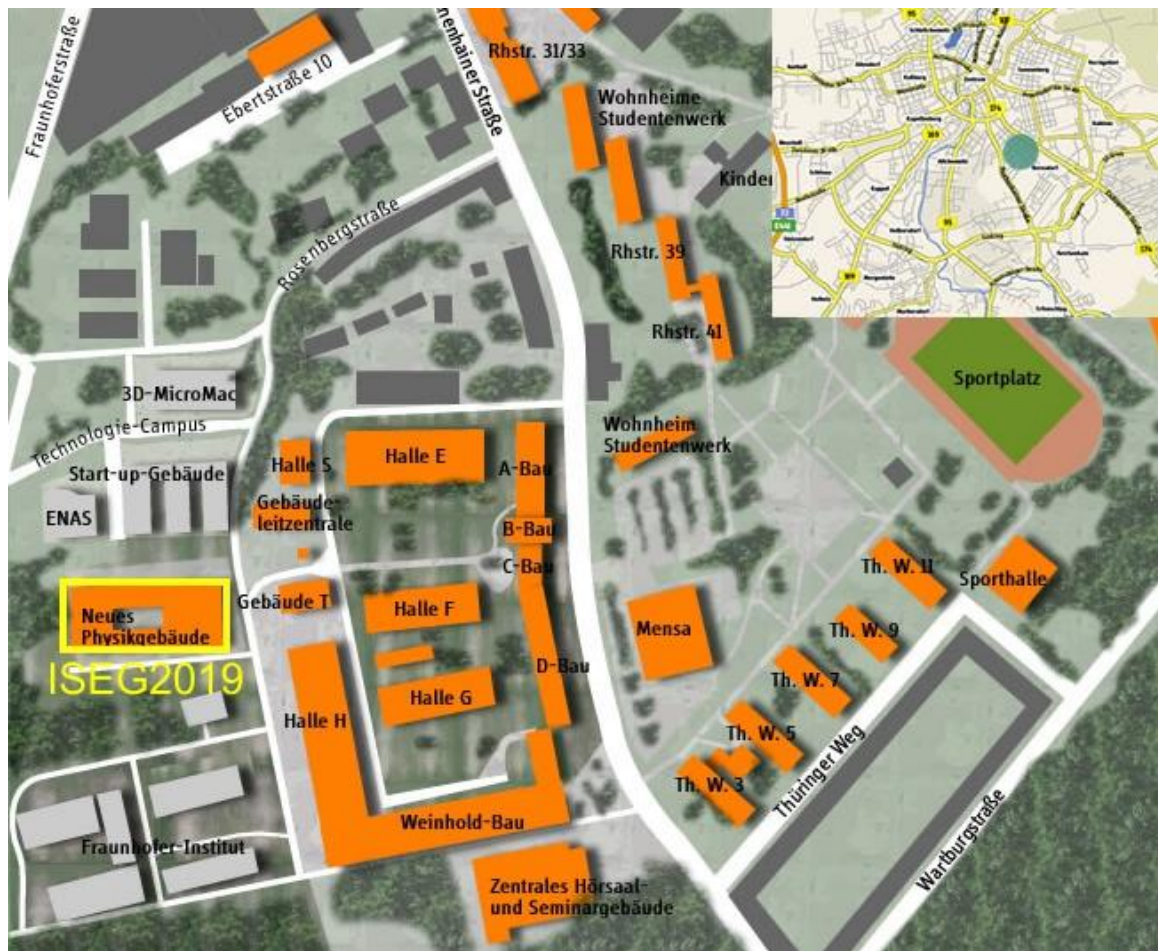
TU Chemnitz

Institut für Physik(Physics building shown on the cover)

Reichenhainer Str. 70

09126 Chemnitz

Rooms P031, P032, P033, and foyer



## Emergency ☎

+49 176 28371883 (Christoph Tegenkamp, mobile)

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## Information for Presenters

- Invited talks are (35+10) minutes.
- Contributed Oral presentations are (25+5) minutes.
- Posters sessions are expected to last about 90 minutes.

## Program Overview

Sunday, 25 August 2019

16:00: Registration and Get-Together

Monday, 26 August 2019

8:45-9:00		Opening remarks	
9:0-9:45	I	<b>Jobst</b>	Intrinsic stacking domains in graphene on silicon carbide: A pathway for intercalation
9:45-10:15	O	Sinterhauf	Electronic transport in graphene on Ge devices on a local scale
10:15-10:45		Coffee break	
10:45-11:15	O	Heilmann	Influence of the buffer layer in epitaxial graphene on the van der Waals epitaxy of h-BN
11:50-12:20	O	Power	Segregated transport channels in sidewall nanoribbons
11:45-12:30	I	<b>Seneor</b>	Introducing 2D Materials for Magnetic Tunnel Junctions
12:30-13:30		Lunch	
13:30-14:15	I	<b>Iacopi</b>	Epitaxial graphene on 3C-SiC on silicon: challenges and perspectives
14:15-14:45	O	Vagin	Electrochemical electroplating/stripping of metals on graphene monolayer: towards silicon carbide based anodes in rechargeable lithium-ion batteries
14:45-15:15	O	Nhung Nguyen	Tuning the electronic properties of epitaxial graphene by molecular self-assembly
15:15-15:45		Coffee break	
16:45-16:15	O	Pradeepkumar	Electrical characteristics of epitaxial graphene on silicon
16:15-17:00	I	<b>Wenderoth</b>	Transport in epitaxial graphene on the nanoscale
17:00	P	Postersession	Aprojanz, Feijo, Gruschwitz, Krone, Kunc, Sakakibara, Schulze, Takeuchi

Tuesday, 27 August 2019

9:00-9:45	<b>I</b>	<b>Ding</b>	Synthesis of Wafer Scale Single Crystalline Graphene by Chemical Vapor Deposition Method
9:45-10:15	<b>O</b>	Schädlich	Properties of epi-graphene grown by PASG on SiC(0001) investigated by low-energy electron microscopy (LEEM)
10:15-10:45		Coffee break	
10:45-11:15	<b>O</b>	Zakharov	Ambivalent behavior of Ge intercalated graphene: interfacial dynamics
11:15-11:45	<b>O</b>	Göhler	Growth and Characterization of Nanocrystalline van-der-Waals Heterostructures on Epi-Graphene Substrates
11:45-12:30	<b>I</b>	<b>Gemming</b>	Calculation of defect- and interface-induced electronic states in 2D materials
12:30-13:30		Lunch	
13:30-14:15	<b>I</b>	<b>de Heer</b>	Epigraphene for graphene based nanoelectronics
14:15-14:45	<b>O</b>	Gerstmann	Bias-controlled doping of graphene with ice: a theoretical DFT study
14:45-15:15	<b>O</b>	Wells	Low temperature graphene-dielectric-semiconductor growth
15:15-15:45		Coffee break	
15:45-16:15	<b>O</b>	Tanaka	Replica of the Dirac cone observed in the ARPES spectra of the epitaxial graphene on SiC
16:15-17:00	<b>I</b>	<b>Norimatsu</b>	Functional epitaxial graphene grown by thermal decomposition of carbides
17:00	<b>P</b>	Postersession	Dedic, Fukuma, Karakachian, Kunc, Reihon, Sato, Schwarz, Uotani



### Wednesday 28 August 2019

9:00-9:45	I	<b>Pierz</b>	Fabrication and properties of ultra-smooth epitaxial monolayer graphene on SiC
9:45-10:15	O	Lin	Towards Tuning the Lattice Orientation of Graphene during Epitaxial Growth
10:15-10:45		Coffee break	
10:45-11:15	O	Imamura	Moire induced electronic structure of twisted bilayer graphene
11:15-11:45	O	Karakachian	Tuning the doping level of graphene in the vicinity of the Van Hove singularity via Yb intercalation
11:45-12:30	I	<b>Tzalenchuck</b>	Polymer-encapsulated molecular doped epigraphene for quantum resistance metrology
12:30-12:40		Closing remarks	
12:40-13:30		Lunch	
14:00-17:00		Excursion	Falconry in castle Wolkenstein
17:00		Conference Dinner	Restaurant "Zum Grenadier" in castle Wolkenstein

**Talks in chronological order – Monday 26.08.2019**

# Intrinsic stacking domains in graphene on silicon carbide: A pathway for intercalation

T.A. de Jong<sup>1</sup>, E.E. Krasovskii<sup>2</sup>, C. Ott<sup>3</sup>, R.M. Tromp<sup>4,1</sup>, S.J. van der Molen<sup>1</sup>, J. Jobst<sup>1</sup>

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Graphene on silicon carbide (SiC) bears great potential for future graphene electronic applications because it is available on the wafer scale and its properties can be custom tailored by intercalating various atoms into the graphene/SiC interface. The intercalation of hydrogen is widely used to convert the buffer layer to quasi-freestanding graphene [1], but more exotic effects such as pn-junctions [2,3] or superconductivity [4] could be observed by intercalating heavier atoms. It remains unclear, however, how atoms can cross the impermeable graphene layer and spread at the interface during the intercalation process.

Here we demonstrate that graphene layers grown in an argon atmosphere on SiC are not as homogeneous as commonly assumed. Instead, epitaxial graphene is intrinsically composed of domains of different crystallographic stacking that are clearly visible in dark-field low-energy electron microscopy (LEEM) [5]. Using quantitative LEEM spectroscopy and first-principles calculations, we show that these domains are the AB and AC versions of Bernal stacking. During growth, two types of domains are formed that can be distinguished by their shape. Nucleation-induced domains have irregular shapes, while triangular domains are formed to accommodate the growth-induced strain between the buffer layer and the first graphene layer or between adjacent graphene layers.

Dislocation lines, in which the strain between the layers is concentrated, are formed between domains. We show that the resulting dislocation networks dominate the (de)intercalation dynamics of hydrogen in quasi-freestanding graphene. Tailoring the dislocation networks, e.g. through substrate engineering, will thus increase the control over the intercalation process and could open a playground for topological and correlated electron phenomena on the wafer scale [6,7].

## References:

- [1] C. Riedl, et al., Phys. Rev. Lett. 103 (2009) 246804.
- [2] K.V. Emtsev, et al., Phys. Rev. B 84 (2011) 125423.
- [3] J. Baringhaus, et al., Sci. Rep. 5 (2015) 9955.
- [4] R. Nandkishore, et al., Nat. Phys. 8 (2012) 158.
- [5] T.A. de Jong, et al., Phys. Rev. Mater. 2 (2018) 104005.
- [6] D. Marchenko, et al., Sci. Adv. 4 (2018) eaau0059.
- [7] SS. Sunku, et al., Science 362 (2018) 1153

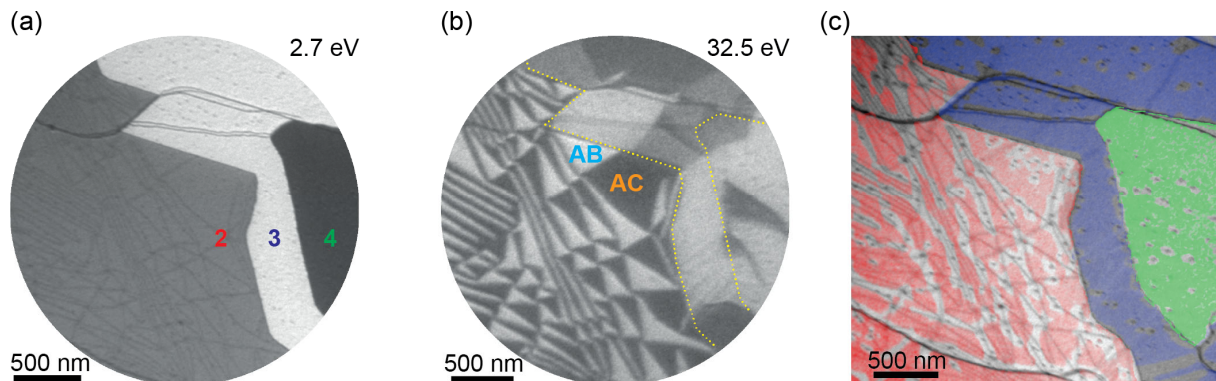


Fig. 1: (a) In Bright-field LEEM, quasi-freestanding graphene appears homogeneous in areas of constant layer number (indicated). (b) In dark-field, however, AB and AC stacking domains are clearly identified. (c) Bright-field image during deintercalation coloured in areas where hydrogen is still present at the interface. It demonstrates that hydrogen atoms deintercalate preferentially along dislocations (dark lines) between domains. All images show the same area of the sample.

# Electronic transport in graphene on Ge/Si(001) devices on a local scale

A. Sinterhauf<sup>1</sup>, S. Bode<sup>1</sup>, M. Auge<sup>2</sup>, M. Lukosius<sup>3</sup>, C. Wenger<sup>3</sup>, G. Lippert<sup>3</sup>, H. Hofsäss<sup>2</sup>, M. Wenderoth<sup>1</sup>

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The successful growth of large-scale graphene on Ge/Si(001) [1] is a significant step towards the integration of graphene into silicon technologies. Since the integration of graphene into graphene-based devices necessarily includes graphene-metal contacts [2], the quality of these contacts crucially limits the performance of the device [3]. Hence, an in-depth investigation of metal contacts on graphene on Ge/Si(001) is required.

In this study, we systematically investigate electronic transport in Gold contacted graphene on Ge/Si(001) by combining the AFM-based method Kelvin probe force microscopy (KPFM) with an additionally applied electric field across the sample (Fig. 1). This allows to measure the electrostatic potential on the nanoscale [4] under ambient conditions, i.e. realistic device conditions. In a systematic approach we investigate the quality of the contacts as well as charge transport in the transition region from the contacts to the graphene sheet and the local voltage drop in pristine graphene areas.

We find that the Gold contacts hinder the exceptional transport properties of graphene on Ge/Si(001) by forming a transition region near the contact edges with significantly reduced electronic performance. Additionally, elemental analysis using Rutherford backscattering shows that the contact area is inhomogeneous. An AuGe alloy is formed driven by diffusion with a Gold Germanium ratio of approximately 50:50. Thus, our study shows that even for this simple device significant limitations in the electrical performance need to be considered. This will be particularly relevant in the manufacture of more complex structures.

## References:

- [1] M. Lukosius et al., ACS Appl. Mater. Interfaces 8 (2016), 33786-33793.
- [2] G. Giovannetti et al., Phys. Rev. Lett 101 (2008), 026803.
- [3] K. L. Grosse et al., Nature Nanotechnology 6 (2011), 287-290.
- [4] P. Willke et al., Carbon 102 (2016), 470-476.

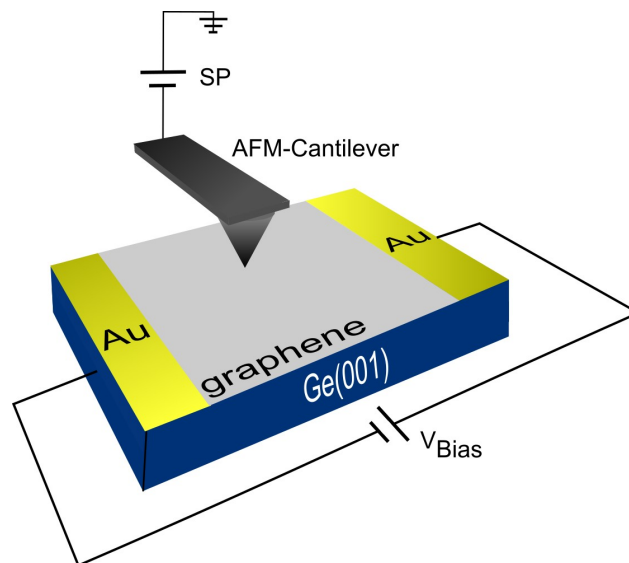


Fig. 1: AFM-based KPFM-setup with additionally applied bias voltage. Graphene on Ge is contacted in a two-terminal geometry and the voltage  $V_{\text{Bias}}$  is applied across the sample for local transport measurements, adapted from [4].

# Influence of the buffer layer in epitaxial graphene on the van der Waals epitaxy of h-BN

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The combination of graphene and its insulating isomorph hexagonal boron nitride (h-BN) into two dimensional (2D) heterostructures allows novel designs for ultra-thin electronic devices, such as capacitors or transistors, comprising of only few atomic layers[1]. However, up to now such heterostructures are mostly fabricated via mechanical stacking of exfoliated flakes, a process which is severely limited in size and throughput. Therefore, van der Waals epitaxy in which the 2D layers are grown on top of each other is currently investigated as a scalable alternative [2,3].

A scalable growth process requires a reproducible substrate material of high quality. In this contribution we present the use of epitaxial graphene as a reliable growth substrate during van der Waals epitaxy of h-BN via molecular beam epitaxy. We have recently already shown that h-BN preferentially nucleates at defects and morphological features on epitaxial graphene on Si-face SiC[4]. Here we investigate an additional influencing factor on the van der Waals epitaxy, which is the buffer layer at the interface between the SiC substrate and the graphene. High resolution transmission electron microscopy (HRTEM) shows the dependence of the growth on the amount of graphene layers, with disordered, multiple layers of h-BN forming on single-layer graphene (SLG), while on bi-layer graphene (BLG) one to two smooth layers of h-BN grew (see Fig. 1). This difference is attributed to a stronger influence of the buffer layer on the growth on SLG, which is screened by an additional layer in BLG. To support this assumption h-BN has been grown on multiple-layers of epitaxial graphene on C-face SiC, where no buffer layer forms at the interface. Furthermore, the buffer layer in epitaxial graphene on the Si-face was transformed into an additional graphene layer via H<sub>2</sub>-intercalation, where h-BN nucleated indistinguishably on the previously SLG and BLG areas. Besides HRTEM the morphology of the samples was investigated via atomic force microscopy, Raman spectroscopy and selected samples via synchrotron-based grazing incidence X-ray diffraction. The contribution highlights the utility and versatility of epitaxial graphene as a substrate for van der Waals epitaxy, which is not limited to the growth of h-BN, but also extendable to other 2D materials, for a scalable fabrication of various 2D heterostructures.

## References:

- [1] K.S. Novoselov et al., Science 353 (2016) aac9439.
- [2] A. Koma, Thin Solid Films 216 (1992) 72.
- [1] J.A. Robinson, ACS Nano 10 (2016) 42.
- [4] M. Heilmann et al., 2D Materials 5 (2018) 025004.

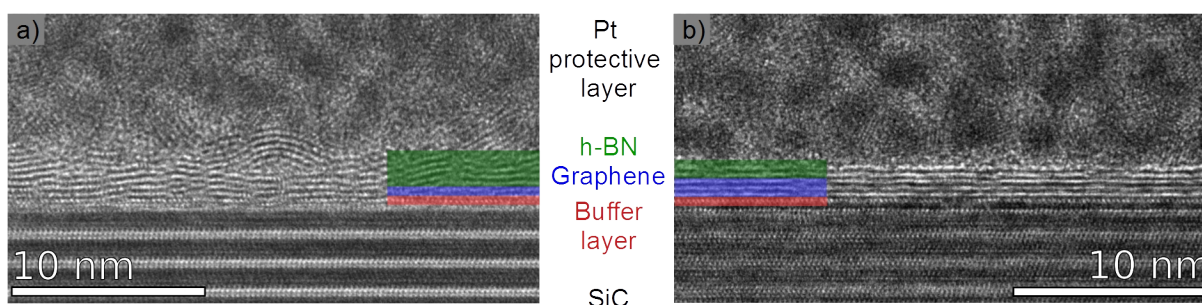


Fig. 1: Comparison of HRTEM images obtained from h-BN grown on (a) SLG and (b) BLG, with electron incidence along SiC(1120).

## Segregated transport channels in sidewall nanoribbons

Stephen R. Power<sup>1,2,3</sup>, Johannes Aproz<sup>4</sup>, Pantelis Bampoulis<sup>5,6</sup>, Stephan Roche<sup>1,7</sup>, Antti-Pekka Jauho<sup>8</sup>, Harold Zandvliet<sup>6</sup>, Alexei Zakharov<sup>9</sup> and Christoph Tegenkamp<sup>4,5</sup>

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<sup>8</sup>CNG and DTU Nanotech, Denmark

<sup>9</sup>MAX IV Laboratory and Lund University, Sweden

Conductance quantization is a defining feature of electronic transport in quasi-one dimensional conductors. In the absence of a magnetic field, confinement results in a sequence of transverse subbands with an increasing number of nodes across the device width.

Graphene nanoribbons grown on the sidewalls of silicon carbide (SiC) mesa structures have previously[1] been shown to present a 1D ballistic channel at the micron scale.

New 2-point measurements reveal additional quantised channels at shorter probe separations[2]. Surprisingly, these channels are localised in different regions across the ribbon width.

Here we demonstrate how this distribution of channels is consistent with a model accounting for both edge zigzag magnetism and asymmetric interfaces between the SiC and nanoribbon at each edge.

References:

[1] J. Baringhaus et al, Nature 506 (2014) 349

[2] J. Aproz, S.R. Power et al, Nature Communications 9 (2018) 4426

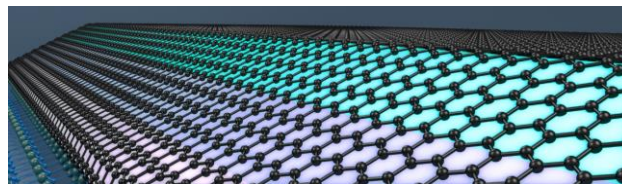


Fig. 1: Electrons in sidewall nanoribbons propagate ballistically, in three separated channels [2].

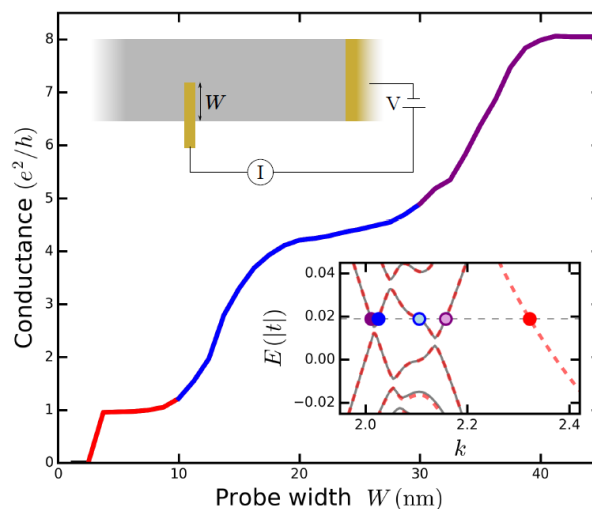


Fig. 2: (Simulation:) The segregated channels lead to quantised conductance steps as one of the transport probes sweeps across the ribbon width [2].

## Introducing 2D Materials for Magnetic Tunnel Junctions

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The recent discovery of graphene, and other 2D materials, has opened novel exciting opportunities in terms of functionalities and performances for spintronics devices. While to date, it is mainly graphene properties for efficient spin transport which have been put forward, we will present here experimental results on another avenue for 2D materials in spintronics. We showed that a thin graphene passivation layer can prevent the oxidation of a ferromagnet and unveil new ALD processes. Importantly, beyond allowing to preserve a highly surface sensitive spin current polarizer/analyzer behavior, the use of graphene on ferromagnets unveiled a new enhanced spin filtering property [1,2]. We will present results concerning 2D materials, from atomically thin insulator h-BN to TMDCs. We will show how ferromagnet metal hybridization can transform an insulating 2D monolayer into a metal [3] and how spin physics may impact 2D based spin valves. These different experiments unveil the promises of 2D materials for spintronics [4] in 2D-MTJ.

### References:

- [1] Dlubak et al. *ACS Nano* 6, 10930 (2012) ; Weatherup et al. *ACS Nano* 6, 9996 (2012)
- [2] Martin et al. *ACS Nano* 8, 7890 (2014) & *APL* 107, 012408 (2015)
- [3] Piquemal-Banci et al. *APL* 108, 102404 (2016) & *ACS Nano* 12, 4712 (2018)
- [4] Review: Piquemal-Banci et al. *J. Phys. D : Appl. Phys.* 50, 203002 (2017)

# Epitaxial graphene on 3C-SiC on silicon: challenges and perspectives

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While epitaxial graphene on 4H or 6H silicon carbide wafers has made extraordinary progress since the pioneering work by Berger and De Heer [1], the process of obtaining EG from silicon carbide on silicon over large areas have turned out to be a much more challenging endeavor [2]. The technological significance of obtaining epitaxial graphene on 3C-SiC on silicon wafers is considerable, and this both from an integration with silicon technology point of view, as for the additional fabrication advantages like the fact that graphene can be obtained in a site-selective way on the wafer –scale [3]. In addition, for nanophotonic applications, where the graphene/SiC material combination could be particularly beneficial, there is a need for optically decoupling array of adjacent devices, which can be more promptly achieved using silicon carbide on silicon.

A lot of the effort over the years has been devoted into try and translate the thermal decomposition route of bulk SiC into a similarly successful approach on 3C-SiC on silicon. While significant progress has been done in terms of fundamental understanding [3, 4] a consistent reproduction over large areas has not been demonstrated to-date using this route, due among others to the exceptionally high defect density characteristic of hetero-epitaxial SiC films. As a result of this limitation, plus a general phenomenon we have recently uncovered causing electrical degradation of the p-n junction at the interface of the 3C-SiC/Si interface [5, 6], no reliable electrical characterization of the transport properties of EG on 3C-SiC on silicon had been possible so far.

We explain how a growth approach making use of a catalytic metal alloy and liquid –phase epitaxy as opposed to the thermal decomposition of the 3C-SiC can help achieving larger –scale and consistent graphene coverage on a highly -defective SiC surface [7]. The consistent graphene coverage, together with appropriate provisions to suppress electrical cross –talk with the substrate has enabled us to perform a systematic evaluation of the transport characteristics of the 2D layer on 3C-SiC on both (100) and (111) silicon substrates. We will share those results, benchmark them with literature data of EG on bulk SiC, and point out current advantages (which include a high adhesion to the substrate), limitations and perspectives. We will also indicate how EG on 3C-SiC is suitable for complementary applications to EG on bulk SiC, where ease of integration is crucial and some level of defectivity can be tolerated or may even be necessary [8].

## References:

- [1] C.Berger et al, Science, May 26; 312(5777):1191-6, 2006.
- [2] *Growing graphene on semiconductors*, ed. Motta, Iacopi and Coletti, PanStanford Publishing, ISBN 9789814774215, Aug.2017.
- [2] B.V.Cunning, M.Ahmed, N.Mishra, A.R.Kermany, B.Wood, F.Iacopi, Nanotechnology 25, 325301, 2014.
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- [8] M Ahmed, B Wang, B Gupta, JJ Boeckl, N Motta, F Iacopi, Journal of The Electrochemical Society 164 (4), A638-A644, 2017.



# Electrochemical electroplating/stripping of metals on graphene monolayer: towards silicon carbide based anodes in rechargeable lithium-ion batteries

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Lithium-ion batteries (LIB) have a supreme conjunction of high energy and power density, which is widely utilized for user electronics, power tools, and electric transportation. Reducing the release of greenhouse gas, the proliferation of LIB technologies is a route towards the energy-sustainable economy. In spite of few decisive advantages, which allowed the LIB commercialization more than 20 years ago, the use of carbon materials as LIB intercalating anodes will become one of the major challenges of a whole LIB technology due to the low specific capacitance of carbon accommodating only 1 lithium atom per 6 carbon atoms. Silicon is very attractive as conversion (alloying) LIB anode material due to its extremely high gravimetric specific capacity with the ratio lithium-to-silicon atoms up to 4:1, low cost, non-toxicity and low lithium discharge potential. However, the dramatic volume change (ca. 370 %) upon lithiation causes fracturing and capacity fading upon charge/discharge cycling. The combination of silicon and carbon as a strategy to marry high capacity and long lifetime is of great interest to the LIB research. Silicon carbide (SiC) in pristine (unmodified) state does not allow the intercalation of lithium due to the close packed lattice, which motivates the strategy to avoid SiC in silicon-based anodes. The thermal annealing of SiC in an inert atmosphere leading to a creation of high quality graphene monolayer residing on top of the reconstructed graphitized SiC surface enables the electrochemical measurements. Firstly, the electrodeposition of model metal (lead) on epitaxial graphene on 4H SiC has been investigated by both experimental and computational approaches [1]. The electrochemical measurements showed the appearance of quasi-reversible electroplating/stripping process of metallic lead on graphene monolayer on SiC. The full agreement of electrochemical measurements, Raman spectroscopy data and computational analysis showed that the lead deposition is much unfavorable on basal defect-free plane of graphene monolayer on SiC in comparison with defect edge plane. Proceeding via instantaneous nucleation mechanism, where the nucleation rate is much higher than the growth rate, the lead electroplating on epitaxial graphene on SiC is characterized with small values of the apparent diffusion coefficient. In coherence with the Raman spectroscopy data and computational analysis, this implies the highly localized non-uniform character of lead deposited species.

Secondly, the electroplating/stripping of lithium on graphitized 4H SiC carried out in the oxygen- and water-free environment showed the slow reversible electrode process yielding the change of the electrode structure. Time-resolved analysis of lithium electrodeposition showed instantaneous nucleation, which is profitable to provide fast and repetitive lithiation/de-lithiation minimizing detrimental dendritic growth that causes the battery failure. The high calculated values of nuclei number density implies the complete and uniform coverage of surface with solid electrolyte interphase (SEI), which confirms the dendritic growth suppression. The high calculated values of apparent diffusion coefficient illustrate a proper mass transport through SEI. Investigated by Raman spectroscopy, the lithiation led to the homogeneous and uniform lithium intercalation featured with disappearance of a buffer layer and the appearance of additional graphene layer. The observed G peak splitting as a result of lithiation suggests the presence of two physically inequivalent graphene layers with different induced doping levels, which might be referred to intermediate intercalation known for graphite.

The reduction of lithium ion, which has the smallest radius, on the epitaxial graphene on SiC yields the uniform and homogeneous intercalation on the contrast to large lead ion yielding highly-localized non-uniform electroplated species. This determines a unique property combination achieved on graphene: out-of-plane permeability for small species with electronic conductivity maintenance. Here, the lithium intercalation into SiC is a result of a pure nanoscale material property: atomic-scale infiltration through the benzene ring of graphene monolayer.

References: These data illustrates the unique

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# Tuning the electronic properties of epitaxial graphene by molecular self-assembly

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Epitaxial graphenes on SiC with tunable work functions are promising templates for a proximity coupling study. Investigation of molecular self-assembly on graphene sheds light to the molecular charge transfer and modifications of the carrier mobility in graphene. We have investigated the adsorption of the non-planar lead-phthalocyanine (PbPc) and magnetic manganese phthalocyanine (MnPc) on n-doped monolayer (MLG) and neutral quasi-free monolayer graphene (QFMLG) by means of scanning tunneling microscopy/spectroscopy (STM/STS), density functional theory (DFT) and in-situ electronic transport measurements. Despite of the fact that both surfaces provide the same lattice constants and symmetry, the adsorption behaviors of phthalocyanines on two substrates are different. PbPc forms up-state islands with a nearly square unit cell on both graphene, but molecular electronic states are strongly shifted with respect to each other, as revealed by STS and DFT. The magnetic MnPc on MLG forms additionally a zig-zag structure together with a normal square one to minimize the adsorption energy. However, the transport data measured on bare MLG and MLG with PbPc on the top proves that neither the charge density nor the mobility of the charge carriers in graphene are not altered.

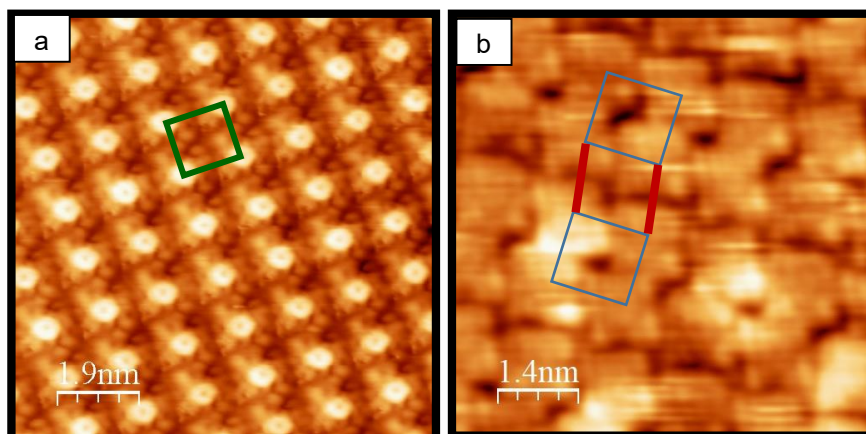


Fig. 1: High resolution STM images reveal (a) the square structure of PbPc (0.1 nA, -1V) and (b) the zig-zag structure of MnPc on MLG (0.4 nA, +2V).

## Electrical characteristics of epitaxial graphene on silicon

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Although major advancements have been achieved in the growth of epitaxial graphene on 4H and 6H SiC wafers and establishment of its transport properties [1-3], an equivalent progress using heteroepitaxial 3C-SiC on silicon with plausible electrical transport measurements has not been fully demonstrated to date. The limiting factors have been not only a non-uniform coverage of graphene on 3C-SiC/Si, but also the instabilities of 3C-SiC/Si heterojunction due to the diffusion of carbon into silicon matrix forming electrically active interstitial carbon defects upon the high temperature heteroepitaxial growth of 3C-SiC, hampering electrical analysis [4].

By addressing the above-mentioned challenges of 3C-SiC/Si interface and non-uniform graphene coverage, in this work we isolate and present the electrical and transport properties of epitaxial graphene (EG) grown directly on cubic silicon carbide on silicon over large areas. To our knowledge, these are the first transport measurements of EG on the 3C-SiC/Si heteroepitaxial system. Here, we compare EG grown on Si(100) and Si(111) substrates using room temperature Hall effect measurements on  $1 \times 1 \text{ cm}^2$  van der Pauw structures, cryogenic transport measurements, Raman spectroscopy, X-ray Photoelectron Spectroscopy (XPS) and Angle-resolved photoemission spectroscopy. Fig. 1 shows that the electrical properties of our EG on both 3C-SiC/Si(100) and 3C-SiC/Si(111) are remarkably in line with those of EG on bulk-SiC wafers from [3]. We find the 300 K mobility of EG grown on 3C-SiC/Si(111), for sheet carrier concentration of  $\sim 10^{12} \text{ cm}^{-2}$ , is  $\sim 10$ -fold greater compared to the EG on 3C-SiC/Si(100). Additionally, XPS measurements confirm the presence of buffer layer only in EG/3C-SiC/Si(111). For EG/3C-SiC/Si(100), we believe the carrier mobility is limited by surface phonon scattering, mediated by the defects in the substrate system [2]. These measurements imply that graphene on 3C-SiC(111) is a natural platform for future devices, which may be integrated, with Si-devices for future heterogeneous applications.

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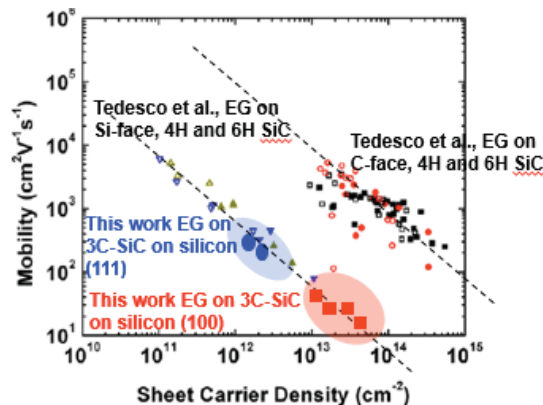


Fig. 1: The sheet carrier densities versus mobilities of EG/3C-SiC/Si from this work are in-line with those of EG on bulk-SiC wafers from Tedesco et al. [Reprinted with permission from AIP]

# Transport in epitaxial graphene on the nanoscale

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The transport properties of epitaxial graphene have been subject of intense theoretical and experimental investigations over the past years. Besides electron-electron and electron-phonon scattering, the charge transport is determined by structural defects such as impurities, substrate steps or monolayer/bilayer junctions. The latter induce spatially varying potential landscape as well as an inhomogeneous current density.

Scanning Tunneling Microscopy combined with a potentiometric extension, called Scanning Tunneling Potentiometry (STP), has opened a way to study these transport properties down to the nanometer scale [1]. Using an STP setup (Fig. 1) based on a home-built low-temperature STM operating down to 6 K and applicable magnetic field of up to 6T, we have investigated the sheet resistance of graphene on the local scale and have analyzed charge transport across different localized defects on a sub-nanometer scale [2]. We find that the voltage drop at a monolayer-bilayer boundary in graphene clearly extends spatially up to a few nanometers into the bilayer and hence is not located strictly at the structural defect. We explain this behavior by the weak coupling between the two bilayer sheets. From magneto-transport STP measurements mapping the local electrochemical potential as a function of the applied magnetic field, we have extracted the local charge carrier concentration by the emerging Hall field. Additionally, we show that the defect resistance at local defects such as steps, wrinkles and ML/BL-junctions remains constant for all magnetic fields applied here.

To determine local resistances quantitatively, the local electric field as well as the local current density are needed. While STP measures the local chemical potential with high precision, the local current density is a priori unknown. In all STP studies up to now, the local current density is replaced by an averaged value, e.g. given by the total current and the geometry of the sample. Graphene grown on 6H-silicon carbide (0001) prepared by polymer assisted sublimation growth (PASG) is characterized by a high degree of a spatial homogeneity [3] resulting in a homogeneous current density. This allows analyzing transport properties quantitatively on the nanometer scale. We demonstrate this new possibility by determining the sheet resistance as a function of the stacking sequence of 6H-SiC. At 8 K, highly resolved STP measurements show a significant variation of up to 270% demonstrating the strong influenced of the underlying substrate on the local transport properties.

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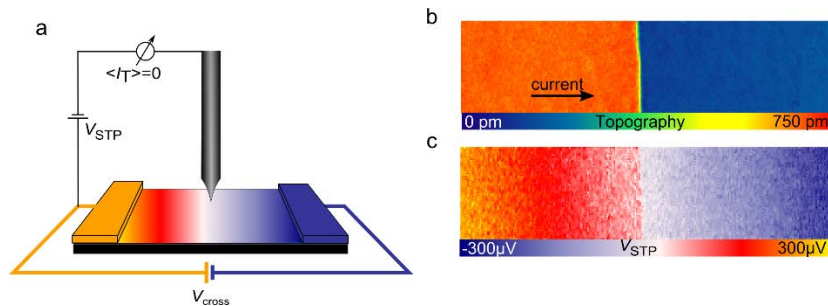


Fig. 1: (a) Working principle of our STP setup: A graphene sample is contacted in two-terminal geometry and a voltage  $V_{cross}$  is applied across the sample. The voltage  $V_{STP}(x, y)$  is adjusted such that the net tunnel current  $I_T$  vanishes. It is recorded at every position of the topography and represents the electrochemical potential of the sample at the position of the tip. (b)  $(200 \times 50) nm^2$  topography (imaging conditions:  $V_{Bias} = 0.03V, I_T = 0.15 nA, j = 3.56 Am^{-1}$ ) of monolayer graphene crossing a triple substrate step and (c) simultaneously recorded potential map.

**Talks in chronological order – Tuesday 27.08.2019**

# Synthesis of Wafer Scale Single Crystalline Graphene by Chemical Vapor Deposition Method—From Theoretical Prediction to Experimental Realization

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Wafer scale graphene single crystal is ideal for maximizing the performances of graphene-based devices. Here we demonstrate a few potential routes toward the synthesis of wafer scale graphene single crystal:

- I. Considering the weak van der Waals (VDW) interaction between graphene and various substrates, our theoretical analysis and molecular dynamic simulations showed that a single crystal graphene can across a grain boundary (GB) of a substrate without forming any defect. Therefore, we propose that wafer scale graphene single crystal might be synthesized on a polycrystalline substrate via nucleation suppression. [1] Such a strategy was realized by a method called “feedstock local feeding” CVD growth and 1.5 inch sized graphene single crystals were successfully synthesized experimentally. [2]
- II. The preferential alignment of nucleated graphene islands on a substrate allows us to grow single crystalline graphene on a symmetry-matching substrate. Therefore, wafer scale graphene single crystal might be synthesized on a wafer scale single crystalline substrate. [1,3,4] Experimentally, large area single crystal Cu (111) foils were successfully synthesized and the CVD growth of wafer scale graphene single crystals was demonstrated. [5,6]
- III. Besides the (111) surface of fcc crystals and the (0001) surface of the hcp crystals, our theoretical analysis predicts that unidirectional growth of graphene or hexagon boron nitride on a low symmetric high-index substrate (such as the vicinal surfaces of Cu(110)) is also possible. [7]

Reference:

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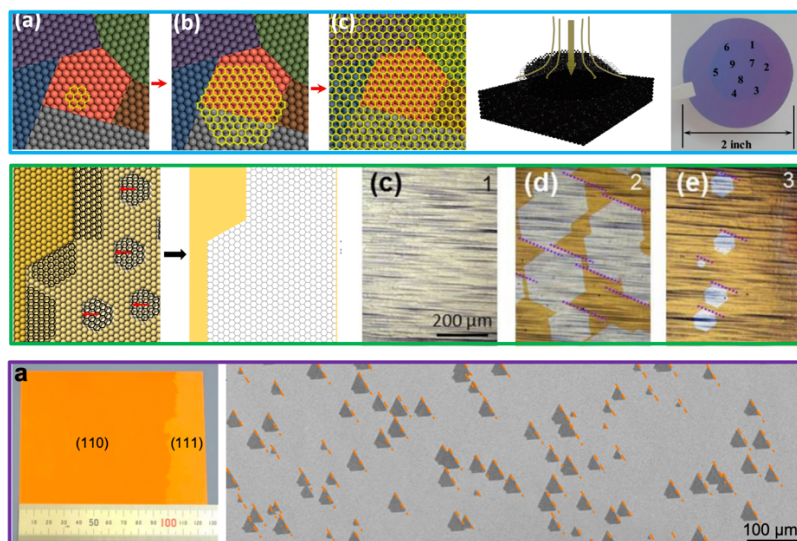


Figure 1. Three potential routes towards the wafer scale synthesis of graphene or hexagonal boron nitride: by single seed nucleation(top); via epitaxial growth on Cu(111) surface (middle); and hBN growth on the vicinal Cu(110) surface (bottom).

# Properties of epi-graphene grown by PASG on SiC(0001) investigated by low-energy electron microscopy (LEEM)

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Although much research has been devoted to the growth of graphene by different approaches and on different substrates, still to this day new phenomena are discovered, which need to be understood in order to fully control graphene synthesis. One of the most promising synthesis routes is the epitaxial growth on silicon carbide, which is subject to our present study.

Recently, the synthesis of epi-graphene was improved by the introduction of the polymer-assisted sublimation growth (PASG) [1]. The main difference between the sublimation growth in argon atmosphere [2] and PASG is the deposition of a polymer film onto the SiC substrate prior to the annealing, which acts as an additional carbon source. This results in an enhanced nucleation of the buffer layer and suppressed step bunching, which usually occurs upon the graphene formation by sublimation. Thus, PASG leads to smooth and homogeneous monolayer graphene with minimum step size equivalent to one or two SiC-bilayers. As a result of the latter, various surface terminations of the SiC substrate exist underneath the graphene. In addition, since graphene growth happens in a ribbon-like fashion nucleating at the steps of the SiC substrate, it is possible to achieve isolated stripes of graphene resting on the buffer layer. The uncovered buffer layer in between the graphene stripes can be decoupled from the substrate by hydrogen intercalation [3]. This results in a pattern of monolayer and quasi-freestanding monolayer graphene. The properties of such surfaces are investigated by electron microscopy and spectroscopy.

References:

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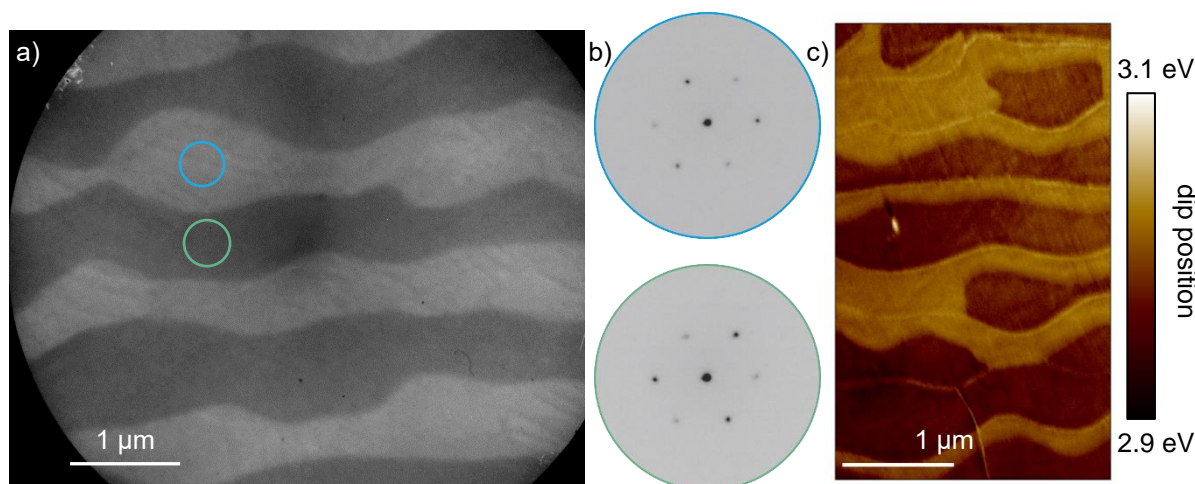


Fig. 1: a) The LEEM dark field image (using a buffer layer satellite spot) of epi-graphene shows a stripe pattern. Bright and dark stripes correspond to surface terminations rotated by  $60^\circ$  to each other. b) Selected area LEED patterns obtained from the positions indicated in a) reveal the rotation of the underlying crystal symmetry for adjacent stripes. c) LEEM image color-coded according to the minimum energy of the electron reflectivity measured in the respective position of the sample.

## Ambivalent behavior of Ge intercalated graphene: interfacial dynamics

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For the realization of graphene-based electronic devices, the functionalization of this material becomes essential. Graphene doping through intercalation and tuning the chemical potential is one of the promising concepts. Intercalation of germanium is particularly interesting in view of its ambivalent doping behavior [1-3]. Both p- and n-type doped graphene and their doping levels were identified by X-ray Photoelectron Emission microscopy (XPEEM), Scanning Tunneling Microscopy (STM) and Angle Resolved PhotoEmission Spectroscopy (ARPES). The absolute amount of intercalated Ge was determined to be roughly 1ML and 2MLs for n- and p-phase, correspondingly. As a template for the intercalated graphene, the buffer layer on SiC(0001) surface was prepared by heating 6H-SiC substrate at high (1700<sup>o</sup>C) temperature in an argon environment. After deposition of ~5ML Ge followed by annealing at the intercalation temperature (700<sup>o</sup>C) to retrieve a p-phase a short post annealing at slightly higher temperatures results in the formation of non-homogeneously (p- and n-type) intercalated areas. Concrete implementation of mutual distribution of p- and n-phases depends on the temperature, mobility of Ge atoms in the second intercalated layer, cooling/heating protocol and can be nicely followed *live* in Low Energy Electron Microscopy (LEEM) during heating/cooling below 500<sup>o</sup>C. The figure below contains three still LEEM images extracted from a movie on cooling (from 500<sup>o</sup>C down to RT) of a Ge intercalated graphene. At 500<sup>o</sup>C (left LEEM image) Ge atoms in the second intercalated layer are mobile in both p- and n-phase (white and black contrast, correspondingly). On cooling, at 300<sup>o</sup>C (middle LEEM image) Ge atom mobility/diffusion length decreases and apparent n-type area breaks in many small p-n junctions. But apparent p-type area is still homogeneous at 300<sup>o</sup>C and breaks into microscopically p-n junctions only after further temperature decrease (right LEEM image, acquired at 100<sup>o</sup>C). The enhanced Ge diffusion can be suitable for tailoring ultra-short junction lengths so that pseudo-spin mismatch can be used in future electronic concepts.

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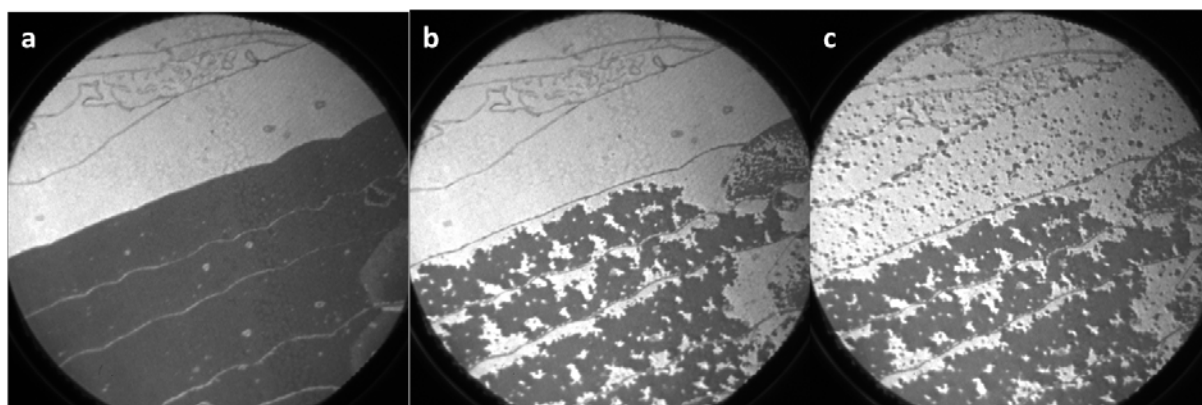


Fig. Three still LEEM images (FoV=25 $\mu$ m, E=4.6eV) of the Ge intercalated graphene at different temperatures: a) T=500<sup>o</sup>C, b) T=300<sup>o</sup>C, c) T=100<sup>o</sup>C. The contrast in the image is due to differently doped graphene (white areas are the p-type graphene and black are the n-type ones). At 500<sup>o</sup>C Ge atoms in the second layer are mobile



# Growth and Characterization of Nanocrystalline van-der-Waals Heterostructures on Epi-Graphene Substrates

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Based on the initial run on graphene and related two-dimensional materials, in recent years we have seen increasing interest in the design of vertical stacks of 2D sheets into so-called van-der-Waals heterostructures [1]. Epitaxial graphene on silicon carbide is an ideal substrate material for this endeavor, as it provides high-quality, wafer-scale graphene resting on a semiconducting substrate as a base for many potential applications for designed structures grown on top.

A rather unique approach to grow precisely stacked multilayer films on a wafer scale is the self-assembly of designed precursors, which is known as the Modulated Elemental Reactants (MER) synthesis [2]. This method applies a two-step process, where first an amorphous precursor with a layering sequence mimicking that of the target compound is deposited at room temperature. This precursor is then crystallized by annealing at moderate temperatures. Due to the low temperatures used during the synthesis, a large number of metastable materials can be created. The individual layers are aligned parallel to the substrate but show random rotational disorder of nanocrystalline domains. Hence, the term “ferecrystal” (based on the latin *ferē* = almost) is commonly used to describe these materials.

Recent research efforts have taken the MER synthesis from thin films towards the 2D limit. In a first exploratory study, we were able to successfully grow a one monolayer thick, nanocrystalline film of MoSe<sub>2</sub> on top of epitaxial graphene on 6H-SiC (0001), by annealing an amorphous precursor to 450 °C as shown in Fig. 1 [3]. The as-grown films were thoroughly investigated by various methods, including X-ray photoelectron spectroscopy, low-energy electron diffraction and Raman spectroscopy. Aside from a small charge transfer between the layers, the structure and properties of underlying graphene layer are conserved during the growth process. Building on these promising initial results, further studies are underway to (a) enhance crystallinity of the as grown films towards wafer scale, monocrystalline layers, and (b) grow more complex 2- or 3-layer stacks of different two-dimensional sheets on epi-graphene substrates.

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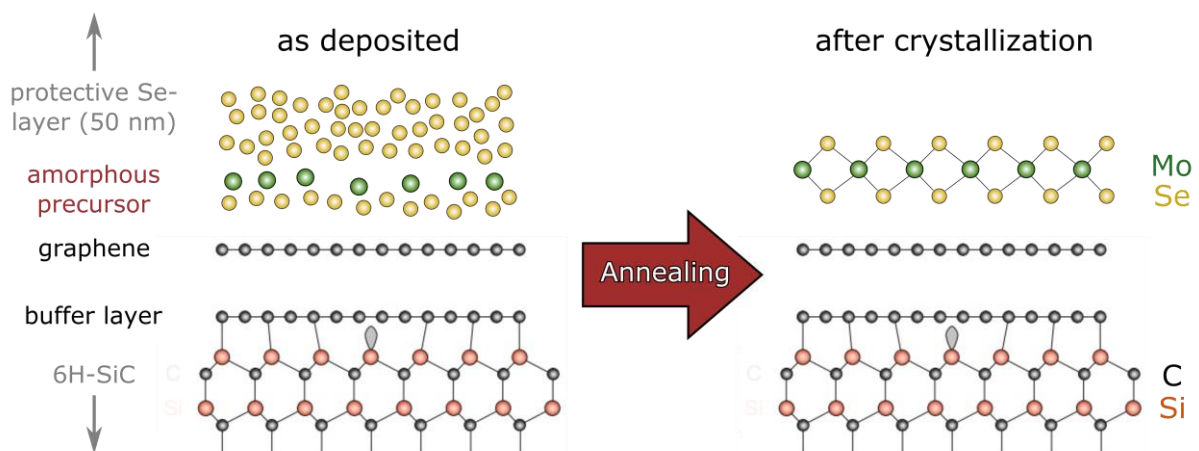


Fig. 1: Schematic growth mechanism for the system epi-graphen – MoSe<sub>2</sub>. Upon annealing at 450 °C at elevated pressure, the deposited amorphous precursor self-assembles into a nanocrystalline monolayer of MoSe<sub>2</sub> resting on the graphene substrate. Reprinted with permission from Ref. [3], © 2018 WILEY - VCH Verlag GmbH & Co. KGaA, Weinheim.

# Calculation of defect- and interface-induced electronic states in 2D materials

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Two-dimensional (2D) materials feature exceptional electronic and optoelectronic properties controlled by the strong confinement in the third dimension. Here, we present calculations within the framework of density functional theory (DFT) to assess the change of 2D materials and their properties under the influence of deviations from the purely 2D nature.

Significant changes to the electronic and optical properties have been monitored already in free-standing 2D layers when comparing structurally perfect monolayers and slightly thicker multilayer structures of the same material, although the multilayer still obeys the same ideal 2D periodicity as the monolayer does. This effect becomes more pronounced once the in-plane symmetry is reduced by rotational stacking faults between the layers of a single 2D material, in van-der-Waals bound heterostructures with other 2D materials, or in the proximity of the substrate. If the adjacent 2D crystal lattices are (nearly) commensurate, such structures still obey periodic boundary conditions in-plane, but with larger superlattice vectors. In that case, the electronic structure undergoes additional modulations within the supercell, which are then periodically repeated in 2D. From a symmetry point of view, the decoration of 2D materials with two-dimensionally periodic assembled organic films may lead to very similar lateral superlattice features, although the interaction of the 2D layer and the individual molecules of the film is local. That provides the possibility to use molecular functionalization for enhancing or suppressing such superlattice features in a predefined way. Finally, decoration can also be employed to heal local structural and electronic defects, which occur depending on the synthesis conditions and break the ideal 2D periodicity of realistic samples. Electronic confinements along 1D boundary lines or localized states at intrinsic defects on the faces cause rather strong local and non-periodic changes of the 2D properties. Calculations suggest that in the limit of low defect density, i.e., below the percolation threshold, the long-range properties of such systems still maintain their 2D nature, but additional effects, such as scattering may result from the interaction with the defects. Molybdenum sulfide is a well-studied binary 2D material, which exhibits all those effects without the need to add additional elements, e.g. hydrogen, to saturate dangling bonds at termination sites. The implications for other, electronically more complex materials such as graphene will be discussed.

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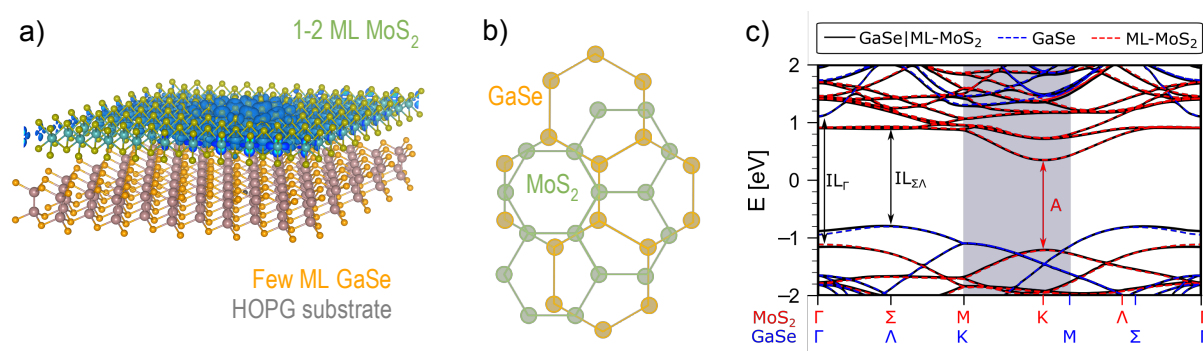


Fig. 1: a) Stacking sequence in the interlayer region between MoS<sub>2</sub> and GaSe, which gives rise to additional interlayer excitonic states with the hole in the GaSe layer. b) Both materials form a nearly commensurate superlattice at a rotation angle of 30°. c) The MoS<sub>2</sub> layer is polarized by the proximity of the GaSe layer. As a consequence, the conduction band edge of MoS<sub>2</sub> is modified and additional states in the band gaps of the pristine materials are obtained.

## Epigraphene for graphene based nanoelectronics

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Epitaxial graphene, or epigraphene, research has been motivated primarily by its promise to succeed silicon electronics in the key areas: speed, energy efficiency, and device density. These extremely lofty goals, which propose to challenge the most highly developed technology in the world, clearly will take a long time to realize. From the outset, the pioneering work of the Georgia Tech epigraphene program (that preceded exfoliated graphene by several years) has focused exclusively on this goal and has had many successes. It is the only graphene electronics platform that adheres to the most basic requirements for a viable nanoelectronics platform: it must be based on a single crystal substrate, and the processes must be scalable. These conditions are required for ultra-high scale integration and reproducibility, as clearly demonstrated in the miraculous evolution of silicon electronics in the past 70 years.

In fact, the lack of epitaxy ultimately why exfoliated nanopatterned devices are insulators, while epigraphene nanostructures can be ballistic on the micron scale, even though the edges are not crystallographically perfect.

In this talk I will present the development of epigraphene on the non-polar faces of SiC, which has edge state transport properties that closely resemble those of the graphene nanoribbons grown on the sidewalls of trenches etched in silicon carbide. The electronic transport is dominated by the single channel edge state with a mean free path exceeding 15  $\mu\text{m}$ , about 1000 times greater than that of the graphene layer. An anomalous quantum Hall effect involving the edge state is observed.

The non-polar epigraphene platform allows interconnected nanostructures to be conventionally patterned to produce 1D networks. At low temperatures, there is clear evidence for Fabry-Perot oscillations, indicating phase coherence over large distances that may be used in future interconnected phase coherent devices. Even though the physics of the transport is not fully understood, it is clear that this discovery provides a unique and feasible route to non-conventional graphene nanoelectronics.

## Bias-controlled doping of graphene with ice: a theoretical DFT study

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Since the discovery of graphene the adsorption of water has been discussed as a promising possibility for doping [1], while changing the electron mobility surprisingly little. Theoretical studies have shown however, that the electronic properties of the resulting system strongly depends on the microscopic details of the substrate [2]. On the other hand, in particular if adsorbed on surfaces, water can occur in many crystalline ice phases [3]. In this theoretical work we investigate the influence of the different ice phases on water adsorption on graphene using density functional theory (DFT).

Taking into account van-der-Waals interaction, we demonstrate that adsorption of a few layers of ice on graphene, free standing as well as adsorbed on silicon carbide, can indeed result in a charge transfer between the ice and graphene layers. In principle, it thus can lead to a doping of graphene. The sign of the charge transfer and by this the type of doping, however, essentially depends on the orientation of the water molecules, whereby the respective differences in total energy are almost negligible. As a consequence, in thermal equilibrium a mixture of different phases is expected, resulting in a cancellation of the doping effect. We further demonstrate that external electric fields can be used to discriminate between different ice phases. Depending on the sign of the bias voltage an electric field component parallel to the surface normal proposes a well-defined control of the doping covering both types of doping, *n*-type as well as *p*-type.

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## Low temperature graphene-dielectric-semiconductor growth

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Problems associated with the preparation of high-quality and contamination-free graphene on semiconductor that does not heavily interact with its underlying substrate has so far hindered its large-scale integration in device structures. We will present our recent work on growing graphene on silicon carbide (SiC) by means of various transition metal catalysts, with subsequent intercalation of silicon and oxygen under the graphene layers to form an electrically insulating dielectric layer, and removal of the transition metal catalyst [1,2]. The basis of this method involves the reaction of thin metal films with SiC [3], such that a metal silicide forms with the liberation of carbon (forming graphene) at industrially viable temperatures of approx. 600°C. The metal silicide layer can be removed by further annealing. Subsequent intercalation of silicon and oxygen results in the decoupling of the graphene from its substrate by forming an insulating silicon oxide layer. The result is highly crystalline top layers of quasi-free-standing graphene, where the thickness of the interfacial dielectric layer can be tuned by controlling the intercalation of silicon prior to the oxidation step.

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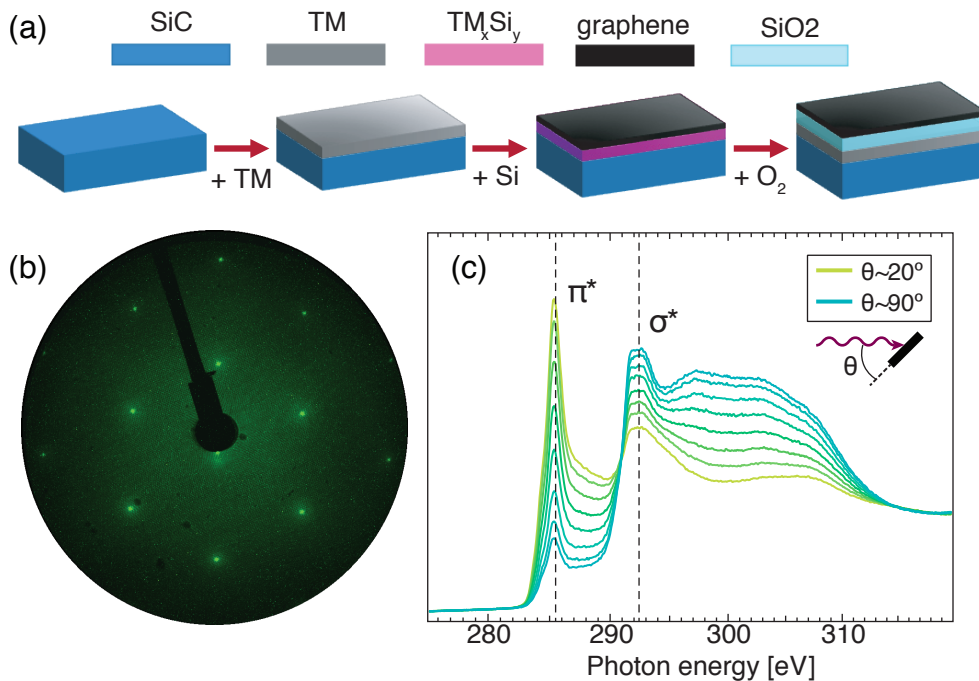


Fig. 1:(a) The preparation of electrically decoupled graphene on semiconductor: 6H-SiC(0001) is coated with 1 nm of a transition metal (TM) and annealed to ~600° C to form graphene, before Si and O is intercalated to form a decoupling layer of SiO<sub>2</sub>. The TM silicide can also be removed (not shown). (b) Diffraction pattern of surface graphene, recorded using low-energy electron diffraction (LEED) at 110 eV. © Angle-dependent NEXAFS measurements in the vicinity of the carbon K-edge, after forming graphene and before SiO<sub>2</sub> intercalation.

# Replica of the Dirac cone observed in the ARPES spectra of the epitaxial graphene on SiC.

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The interaction between the epitaxial graphene layer and the substrate is one of the key issues to understand/control the properties of the epitaxial graphene. In this paper, we investigate the formation of replicas of Dirac cone for the epitaxial graphene caused by the SiC substrate by using angle-resolved photoelectron spectroscopy (ARPES).

All experiments were carried out at HiSOR-BL9A at Hiroshima University. Figure 1 shows the ARPES intensity map (in  $\sqrt{I}$  scale where  $I$  denotes the intensity) at the Fermi level from the graphene/SiC taken at  $h\nu=33\text{eV}$ . All Dirac cones at the K-points are surrounded by six replicas, which are produced by the photoelectron diffraction by the  $(6\sqrt{3}\times 6\sqrt{3})R30^\circ$  periodicity of the “buffer layer” that is formed between the graphene layer and SiC substrate as in previous studies[1]. On the other hand, when the photon energy is near 11eV, completely different replicas appear. In Fig.2, intensity map similarly obtained as Fig.1 except for  $h\nu=11.4\text{eV}$  is shown in linear scale, where several replicas are clearly observed. Note that the scale of the momenta is half of the Fig.1, and there are no true photoelectron emission from the Dirac cone at the K-point. The intensity of the replicas strongly depends on the photon energy; it is of a Lorentzian peak shape with the width of 1.6eV at  $h\nu=11.0\text{eV}$ . The main replicas forming a hexagonal shape may possibly be attributed to the photoelectron diffraction caused by the  $\sqrt{3}\times\sqrt{3}$  lattice of the SiC substrate, and other weak replicas are due to the multiple diffraction. One peculiar thing that should be pointed out is the intensity distribution around the Dirac point; while these show a six-fold symmetry in the case of the original Dirac cone (Fig.1), those of the replicas observed here show two-fold symmetry (Fig.2). This behavior is interesting since it was claimed that the intensity distribution of the photoelectron from the Dirac cone is related to the “phase” term  $\exp(i\theta_q)$ , where  $\theta_q$  is the angle around the K point in the reciprocal lattice space, in the wavefunction of the graphene valence band [2]. We will discuss the origin of this behavior with the help of the result of the photon-polarization-dependent measurement.

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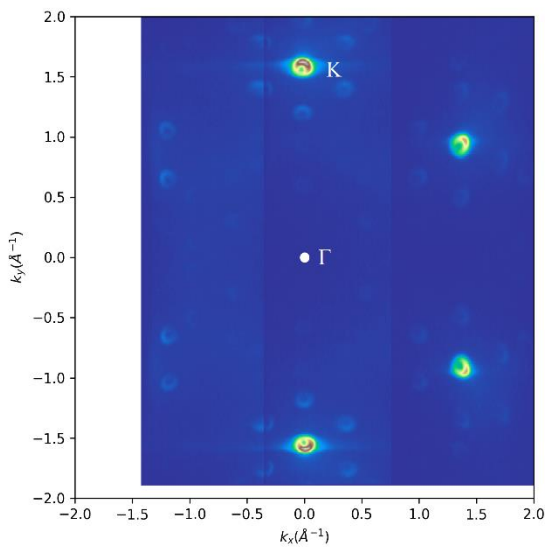


Fig.1 : Intensity map at the Fermi level obtained by the ARPES measurement for the graphene/SiC at  $h\nu=33\text{eV}$ .

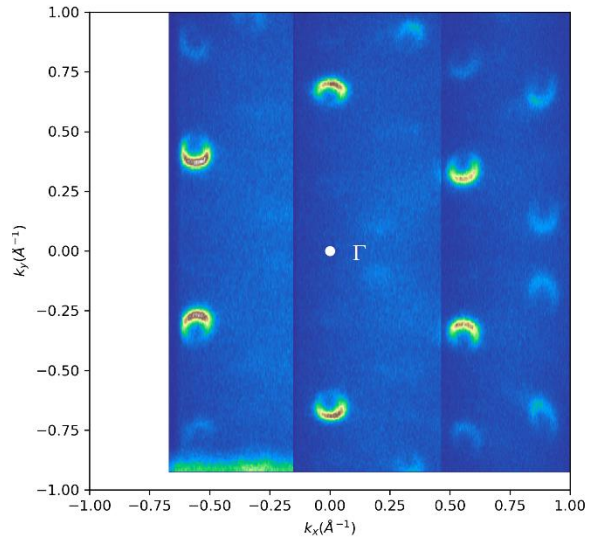


Fig.2 : Intensity map (in linear scale); similar to Fig.1 but taken at  $h\nu=11.4\text{eV}$ .

# Functional epitaxial graphene grown by thermal decomposition of carbides

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Epitaxial graphene growth on SiC is the only technique to grow wafer-scale single-orientation graphene directly on the insulating substrate. This technique utilizes the feature that carbon tends to remain on the SiC surface during decomposition as a solid state compared with silicon. It suggests the possibility that we can grow graphene by thermal decomposition of other carbides. We have shown that graphene could be actually grown by thermal decomposition of titanium carbide thin film and boron carbide powder [1,2]. In this talk, I will present recent studies about graphene growth by thermal decomposition of carbide thin films, such as aluminum carbide ( $\text{Al}_4\text{C}_3$ ), boron carbide ( $\text{B}_4\text{C}$ ), and tantalum carbide ( $\text{TaC}$ ), which is epitaxially grown on SiC.

Carbide thin film was deposited on the SiC surface by the pulsed laser deposition (PLD) technique. The substrate temperatures were varied from room temperature to 1500 C. The PLD conditions were optimized depending on the target carbides. Graphene was grown by thermal decomposition of carbides under Ar or vacuum.

Figure 1 shows the high-resolution transmission electron microscope (HRTEM) images of graphene/ $\text{B}_4\text{C}$ /SiC [3]. Both  $\text{B}_4\text{C}$  and graphene were grown epitaxially on the substrate with the orientation relation  $[11-20]_{\text{Gra}} // [11-20]_{\text{B}_4\text{C}} // [11-20]_{\text{SiC}}$  and  $(0001)_{\text{Gra}} // (0001)_{\text{B}_4\text{C}} // (0001)_{\text{SiC}}$ . As a result of the magnetic properties measurements, graphene/ $\text{B}_4\text{C}$ /SiC samples exhibited the spin glass behavior at low temperatures, which suggests the presence of local antiferromagnetic ordering in graphene. Similarly, graphene was grown by thermal decomposition of  $\text{Al}_4\text{C}_3$  and TaC. I will also present the results of our attempts to improve the carrier mobility of graphene on SiC [4].

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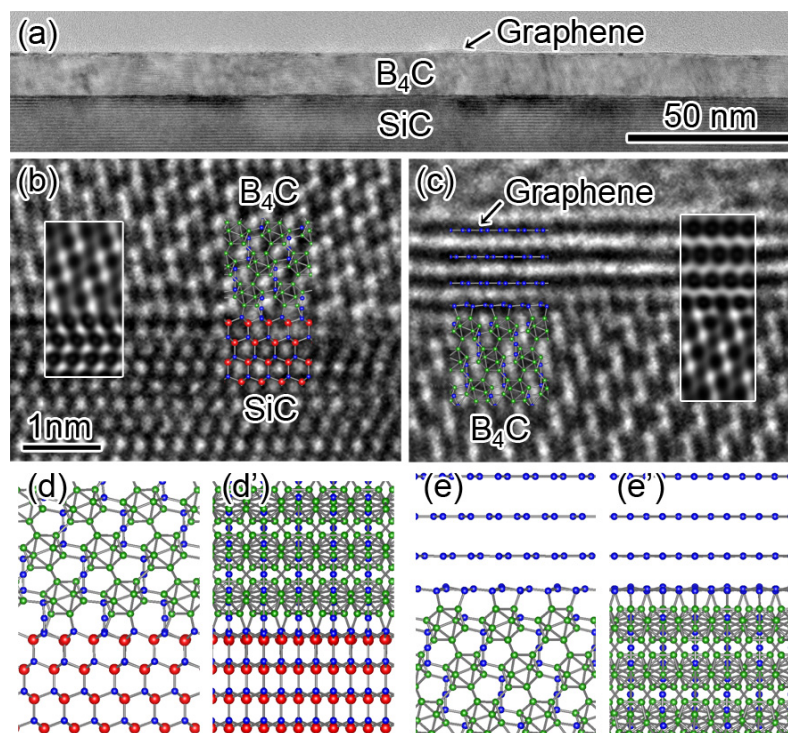


Fig. 1: HRTEM images of graphene/ $\text{B}_4\text{C}$ /SiC. (a) Low magnification image. (b) and (c) HRTEM images of the interfaces of  $\text{B}_4\text{C}$ /SiC and graphene/ $\text{B}_4\text{C}$ , respectively. (d-e) Structural models of the interfaces after structural optimization using density functional theory calculations.

**Talks in chronological order – Wednesday 28.08.2019**



# Fabrication and properties of ultra-smooth epitaxial monolayer graphene on SiC

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Epitaxial graphene growth on SiC (0001) is often accompanied by step-bunching of the underlying SiC terraces and graphene bilayer formation at the step edges. These both deteriorate the homogeneity of the graphene layer and the performance of graphene-based devices. Here we show that rapid buffer layer nucleation is the key to suppress the step-bunching and to obtain homogenous large-area monolayer graphene. Moreover, fine-tuning of a set of growth parameters allows us to control the surface terracing.

The epitaxial graphene was fabricated by polymer-assisted sublimation growth (PASG) in which additional carbon supply by a cracked polymer on the SiC surface leads to a rapid buffer layer nucleation already at temperatures below 1400 °C (1 bar Ar atmosphere) [1]. The large-area buffer layer on the SiC terraces stabilizes the SiC surface, thereby thermal dissociation of SiC layers is reduced and step retraction is slowed even after further temperature rise (~1750 °C) for graphene growth.

In order to control the SiC step structure and the buffer layer formation, we show that the so-far less regarded flow rate of Ar gas during the growth process offers another vital parameter to optimize graphene growth in addition to Ar pressure, substrate temperature and growth time. Fine-tuning of the parameters allows the growth of continuous graphene-free buffer layer sheets which is verified by the high uniformity of the QFMLG and QFBLG sheets after hydrogen intercalation [2].

For the optimized graphene growth, ultra-smooth surfaces can be obtained with 90 % and 70 % of the steps being < 0.75 nm high on 6H- and 4H-SiC substrates, respectively [3]. The low step heights prevent bilayer formation at the terrace edges, which results in very homogenous monolayer graphene sheets on mm scales, verified by Raman mappings. Due to the bilayer exclusion, this pure monolayer graphene sheet exhibits negligible resistance anisotropy values (~ 2%) in both  $\mu\text{m}$ -scale rotational square-four-point probe and mm-scale VdP measurements. In comparison to nanoscale scanning tunneling potentiometry investigations, it can be concluded that the ultimate lower limit of resistance anisotropy is reached which is attributed to unavoidable resistance contributions of the step edges [3].

The stabilization of the SiC surface by rapid buffer layer formation during step retraction results in an intermediate state of the SiC step structure. For 6H-SiC/G, we obtain a characteristic pattern of repeating step pairs with 0.25 and 0.5 nm in height [3]. This is accompanied by a sequential color contrast observed in AFM phase, Kelvin-probe, SEM, and LEEM images which is attributed to an alternating surface potential correlated with unequal surface energies of the underlying SiC stacks. This is inferred from a simple step flow model based on the different retracting speeds of SiC terminations, which enables identification of each of the underlying SiC stacks.

Our results give an insight into the complex buffer and graphene layer synthesis as well as step-bunching of SiC surfaces. We have shown that it is possible to control the terrace structure of SiC/G by means of growth parameters. This suggests an approach for substrate induced doping engineering on the nanoscale of SiC/G or other 2D material systems.

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# Towards Tuning the Lattice Orientation of Graphene during Epitaxial Growth: A Platform to Study Twisted Bilayer Graphene

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There exists a large and still growing variety of 2D materials with a broad range of physical properties. Because 2D materials are held together by van der Waals interactions only, one is relieved from the lattice-match constraint of conventional epitaxy and can create completely new artificial materials (in which the interfaces are the material), not only by stacking arbitrary (monolayer) sequences, but also by carefully controlling the twist angle between the layers [1]. A remarkable example for the emergence of unexpected properties is twisted bilayer graphene, that becomes superconducting at a magic twist angle of  $1.1^\circ$ , and at larger angles exhibits a network of topological channels [2]. Here we present epitaxial methods to control the lattice orientation of graphene single and bilayer.

Single and multiple layers of graphene grown epitaxially on SiC(0001) are known to be rotated by  $30^\circ$  with respect to the SiC lattice ( $R30^\circ$ ). We compare here two routes to force a  $R0^\circ$  orientation upon the lattice of single layer graphene on SiC. This then serves as a platform to further grow  $R30^\circ$  graphene or intercalate the buffer-layer in order to create twisted bilayer graphene. The first route consists of using a single layer of hBN- $R0^\circ$  on SiC as a template for the growth of graphene. The small lattice mismatch between hBN and graphene allows seeds of graphene to appear within the hBN layer and finally graphene fully replaces the hBN layer. An alternative route consists of growing  $R0^\circ$  graphene in an atmosphere of borazine molecule thus using the molecules as a surfactant. The quality of the obtained layers is compared using spot profile analysis low energy electron diffraction, angle-resolved photoelectron spectroscopy and normal incidence x-ray standing wave.

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# Moiré induced electronic structure of twisted bilayer graphene

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Twisted bilayer graphene (TBG) shows variety of electronic characteristics depending on its in-plane rotation-angle due to moiré potential. The Fermi velocity decreases dramatically with the angle less than  $5^\circ$  and moreover, superconductivity is emerged at approximately  $1^\circ$  magic angle [1]. TBG has been fabricated by tape-exfoliation and transferring processes. However, these processes have potential difficulties in obtaining large area, precise control of the rotation angle, and contaminant-free interlayer structures. A large area sample is especially essential to evaluate electronic structure by macro-probe characterizations such as angle-resolved photoemission spectroscopy (ARPES). Thus, we aimed at solving these problems by direct transfer of newly developed CVD grown graphene on SiC in vacuum [2].

A set of monolayer graphene samples was first grown on 4H-SiC(0001) by CVD using  $C_2H_4/Ar$  mixed gas. This CVD graphene can be easily exfoliated due to a  $(3 \times 3)$  buffer layer consisting of Si-O-C, because this  $(3 \times 3)$  buffer layer has much weaker bonding to graphene than  $(6\sqrt{3} \times 6\sqrt{3})R30^\circ(6R3)$  buffer layer normally formed by SiC thermal decomposition [3]. Two samples were appressed face to face in high vacuum at  $\sim 200^\circ C$ , held for  $\sim 1$  hour, and then detached, completing transfer procedure. TBG area on the millimeter scale was confirmed by optical microscopy and Raman spectroscopy. Figure 1(a) shows the low-energy electron diffraction (LEED) pattern from the TBG area. Two sets of graphene spots, being rotated  $4^\circ \pm 0.5^\circ$  with respect to each other, were clearly observed. More importantly, satellite spots originated from the moiré structure were recognized in the vicinity of the graphene spots as shown in Fig. 1(b). ARPES was then performed along  $K_1$ - $K_2$  direction as indicated in Fig. 2(a). Three Dirac cones were identified in Fig. 2(b). Fig. 2(c) describes the origin of these Dirac cones, i.e. CVD graphene  $K_1$ , transferred graphene  $K_2$ , graphene on the 6R3 buffer layer, which inevitably appears in small patches during CVD growth. The energy gap and reduced Fermi velocity were observed where  $K_1$  and  $K_2$  Dirac cones are crossing (van Hove singularity). We will present the results of detailed ARPES analyses on this TBG as well as TBGs with different rotation-angles.

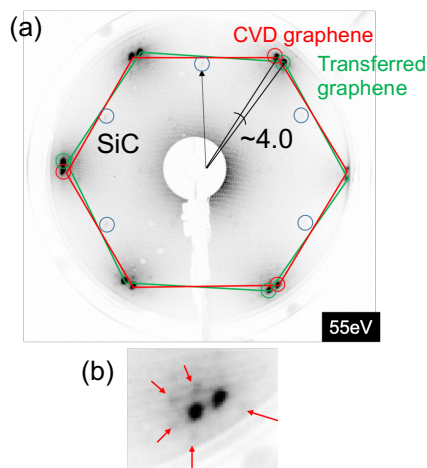


Fig. 1 (a) LEED pattern of  $\sim 4^\circ$  TBG. (b) Magnified image of graphene diffraction spots, showing satellite features due to the moiré structure.

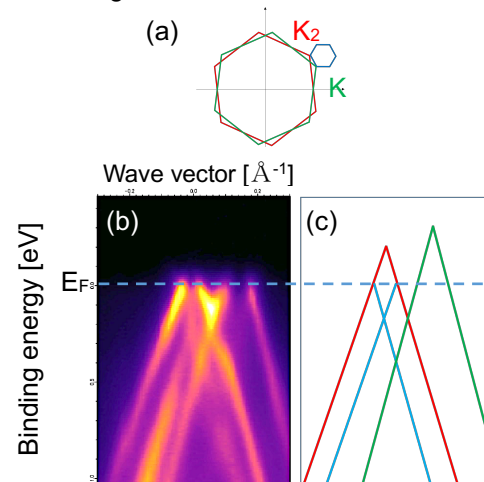


Fig. 2 (a) Brillouin zones of CVD and transferred graphenes and TBG (blue hexagon). (b) ARPES spectra from TBG along  $K_1$ - $K_2$ . (c) Schematic drawing of (b).

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# Tuning the doping level of graphene in the vicinity of the Van Hove singularity via ytterbium intercalation

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In heavily n-doped graphene, when pushing the Fermi level into the vicinity of the Van Hove singularity, exotic electronic ground states are expected to occur driven by many-body interactions [1]. These competing phases find their stability based on the amount of doping induced in the graphene layer. In this work, we present a method for effectively tuning the doping level of graphene near the Van Hove singularity. Epitaxially grown buffer layer graphene on SiC(0001) is decoupled from the SiC substrate and strongly n-doped up to its Van Hove singularity via ytterbium intercalation. Upon annealing the Yb/graphene system at increasing temperatures, a topological transition at the Fermi level and a continuous upshift in Dirac point energy are observed due to a lowered charge carrier density. The intercalated Yb atoms develop an ordered pattern and their concentration changes with annealing temperature. These variations significantly affect the amount of charge transferred to the graphene layer and allow the systematic control of doping level near the Van Hove singularity via one single experimental parameter. As such, the Yb intercalation technique can provide a reliable way of accessing different ordered ground states predicted in highly doped graphene.

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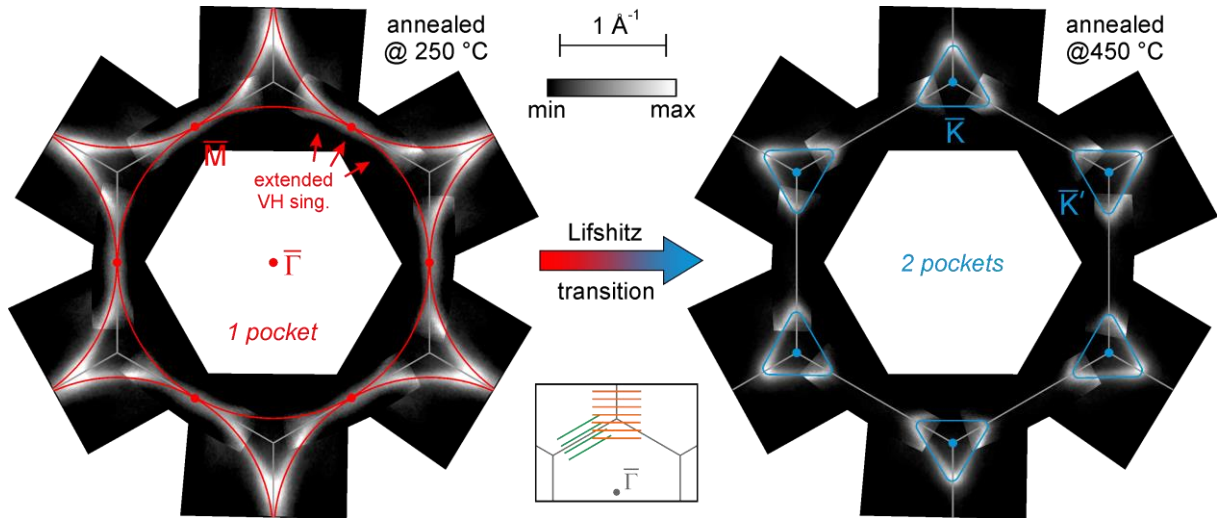


Fig. 1: Fermi surfaces acquired after annealing the sample at 250 °C (a) and at 450 °C (b). The Fermi surfaces are constructed by symmetrizing two sets of energy-momentum cuts taken perpendicular to  $\Gamma K$  (marked in orange in the inset) and parallel to  $KMK'$  (marked in green). The solid red and blue curves are guides to the eye indicating the respective Fermi surface topology.

# Polymer-encapsulated molecular doped epigraphene for quantum resistance metrology

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Effective from May 20, 2019 the international system of units, the SI, is defined in terms of fundamental constants of Nature. Graphene, and epitaxial graphene on silicon carbide (epigraphene) in particular, has played a role in the long process towards this pivotal moment, when used to confirm the universality of the resistance quantum  $h/e^2$ . In the future it will be central to dissemination of the units, including the ampere and the kilogram defined in terms of the elementary charge  $e$  and the Planck's constant  $h$  respectively.

One of the aspirations of quantum metrology is to deliver primary standards directly to end-users thereby significantly shortening the traceability chains and enabling more accurate products. Epitaxial graphene grown on silicon carbide is known to be a viable candidate for a primary realisation of a quantum Hall resistance standard, surpassing conventional semiconductor two-dimensional electron gases (2DEGs), such as those based on GaAs, in terms of performance at higher temperatures and lower magnetic fields. The bottleneck in the realisation of turn-key quantum resistance standard requiring minimum user intervention has so far been the need to fine-tune the carrier density in this material to fit the constraints imposed by a simple cryo-magnetic system. Previously demonstrated methods, such as via photo-chemistry or corona discharge, require application prior to every cool-down as well as specialist knowledge and equipment. To this end we perform metrological evaluation of epigraphene with carrier density tuned by a recently reported permanent molecular doping technique. Measurements at two National Metrology Institutes (NMIs) confirm accurate resistance quantisation below 5 nΩ/Ω [1]. Furthermore, samples show no significant drift in carrier concentration and performance on multiple thermal cycles over three years. This development paves the way for dissemination of primary standards based on epigraphene.

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**Posters in alphabetical order – Monday 26.08.2019**

## Ballistic tracks in graphene nanoribbons

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The electronic confinement in graphene nanoribbons (GNR) leads to the formation of subbands as well as topologically protected edge states. The selective growth by sublimation on nanofacets of SiC mesa structures produces GNR of exceptionally high quality and well-defined edges. Their hallmark is a probe-spacing and temperature independent conductance of  $1 \cdot e^2/h$  which strongly indicates fully spin-polarized ballistic transport. These robust signatures can be observed in a range of a few  $\mu\text{m}$  down to hundreds of nm at room temperature.

Compared to former experiments contacts with even smaller probe spacings, as small as 70 nm, were realized by means of a 4-tip STM/SEM. On these length scales sudden jumps to plateaus of multiples of the conductance quantum  $e^2/h$  were observed using one blunt tip covering the entire GNR and one sharp tip gradually crossing the ribbon. These values refer to fourfold degenerated ballistic channels. We attribute these plateaus to edge and bulk transport channels, respectively. Based on tight-binding calculations the transport findings can be modelled including asymmetric coupling of the ribbon edges resulting in transversal electric fields which originate from distinct edge terminations on both sides of the GNR. Finally, the conductance across the ribbon is calculated reflecting the experimental findings

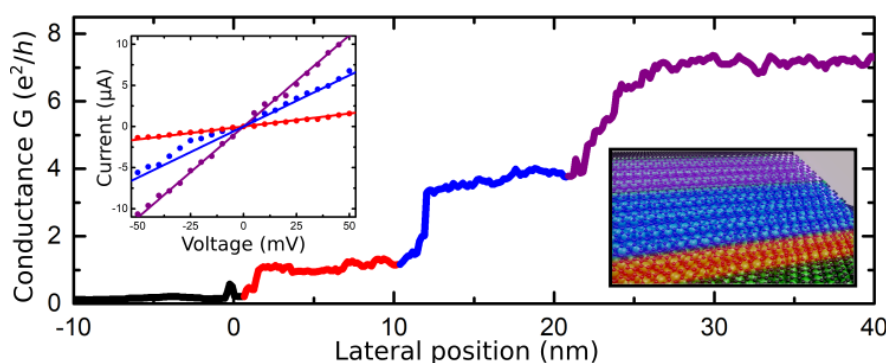


Fig. 1: Conductance  $G$  measured for a fixed distance  $L=70$  nm and bias voltage  $U=200$  mV. Starting from the lower edge the sharp tip was gradually moved across the GNR while the blunt tip was covering the entire GNR. Inset: IV-curves measured at bottom, middle and top of the GNR.

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# Growth of MoS<sub>2</sub> on epitaxial graphene by CVD

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Van der Waals (vdW) heterostructures have captured scientific attention because of their unique electronic and optical properties [1]. Thus it is highly important to investigate the large-area synthesis of such material systems in order to implement them in future applications. In this work, we investigated the growth of MoS<sub>2</sub> by chemical vapor deposition (CVD) (using vapor transport of MoO<sub>3</sub> and S powders) on epitaxial graphene/SiC(0001). Different growth temperatures between 550 to 700 °C were employed. The epitaxial graphene templates were synthesized by surface graphitization of SiC(0001) surfaces at high temperature (1600 °C) in an Ar atmosphere [2]. Additionally, MoS<sub>2</sub> was simultaneously prepared on amorphous SiO<sub>2</sub> (on Si) templates in order to allow for a direct comparison of the samples. The structural characterization of the samples was performed using atomic force microscopy (AFM), X-ray photoelectron spectroscopy, Raman spectroscopy, and synchrotron-based grazing incidence X-ray diffraction (GID). Figures 1(a) and 1(b) show AFM images for MoS<sub>2</sub> grown on epitaxial graphene and SiO<sub>2</sub>. The triangles observed in both images are MoS<sub>2</sub> structures. The MoS<sub>2</sub> grown on epitaxial graphene exhibits a preferential alignment to the SiC surface steps (vertical stripes in the figure). GID confirms the epitaxial growth of MoS<sub>2</sub> on graphene. Furthermore, the MoS<sub>2</sub> islands grown on epitaxial graphene offer improved morphology (e.g. sharper edges, larger sizes) in comparison to those formed on the amorphous substrate. Formation of multi-layer thick MoS<sub>2</sub> is also substantially suppressed when epitaxial graphene is utilized as a template. Figure 1(c) shows Raman spectra with the characteristic peaks of MoS<sub>2</sub>, which are narrower for MoS<sub>2</sub> grown on epitaxial graphene and thus confirms the higher material quality [3]. These results help to understand the growth behavior of MoS<sub>2</sub> on different substrates and illustrate the great potential of epitaxial graphene as template for the scalable fabrication of high-quality MoS<sub>2</sub>/graphene heterostructures.

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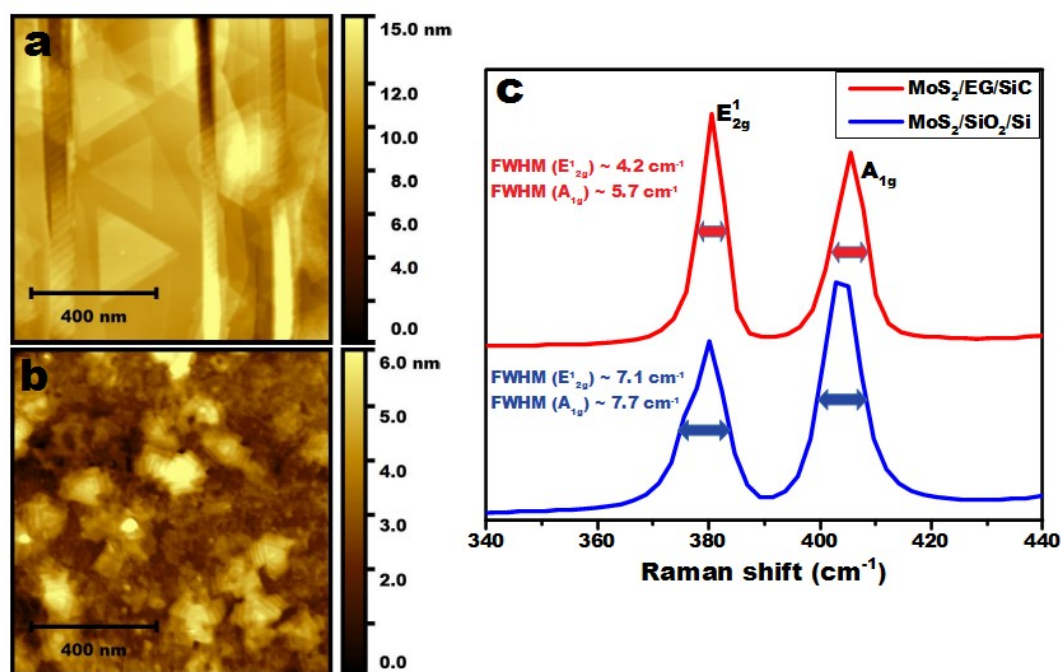


Fig. 1: AFM images of MoS<sub>2</sub> grown on (a) epitaxial graphene and (b) SiO<sub>2</sub>. (c) Raman spectra of MoS<sub>2</sub> grown on epitaxial graphene (red line) and SiO<sub>2</sub> (blue line).



# The microscopic structure of ballistic graphene nanoribbons

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Epitaxial graphene nanoribbons on SiC were shown to host ballistic transport channels, even under ambient conditions. [1] Thereby, the transport along these 40 nm wide ribbons, which are grown via self-assembly on sidewalls of SiC (0001) mesa structures, depends crucially on atomistic details of the structure. [2] Here we present high resolution STEM investigations done on such ribbons.

The focus in research was on epitaxial graphene on structured 6H-SiC. A complete understanding of this mesoscopic system requires investigations of the structure at atomic scale as well as the interactions between the atoms. If the relation between the structure and the outstanding electronic properties is known, a controlled influence on the properties of the mesa structure by tuning the structural properties is possible. This is a big leap towards large scale application in nanoelectronics.

Structural and electronic properties of sidewall nanoribbons compared to epitaxial graphene on SiC (0001) are investigated by STEM. The cross section sample preparation was achieved using a dual beam FIB. A double C<sub>s</sub> corrected ThemisZ STEM from ThermoFisher that was operated at 80 keV provided insights in the atomic structure. The system also allowed spectroscopic analysis by EDX and EELS at atomic resolution with a narrow beam energy dispersion amounting to 0.2 eV.

For ribbons along the [1-100]-direction STM reveals zig-zag edge nanoribbons. [2] Thereby, the lower edge merges into the SiC (0001) terrace giving rise to hybridization and formation of an edge state. This growth mode is confirmed by STEM investigations. Cross sectional STEM reveals freestanding graphene separated from the facet by more than three times the distance of graphene on SiC (0001). Moreover, the EDX and EELS capabilities of the HRTEM were used to characterize the surface near areas in detail. Unlike other groups [3] we conducted depth resolved EELS analysis. Upon growth of graphene by desorption of silicon by high temperature annealing, the topmost three SiC bilayers reveal an increasing silicon deficiency towards the interface. This depth dependent concentration confirms XPS measurements applied at these systems. [4, 5] However, a rise of sp<sup>2</sup>-hybridization has already been observed at a depth of one 6H-SiC unit cell. Compared to the facets, it turns out that these residual imperfections in SiC are more frequent at [11-2n] terraces. Combined with a weakened bonding of surface atoms at the sidewall a premature decomposition at the facet is observed, resulting in selectively growing graphene nanoribbons. The absence of a buffer layer at the facet was confirmed by EELS measurements.

In summary the freestanding character of sidewall graphene nanoribbons could be confirmed by STEM micrographs. We found a gradient decomposition of the substrate due to sample production. The weakened bonding of sidewall atoms allow a premature decomposition at the facet resulting in selective growth.

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## Tracking stepwise hydrogen intercalation of epitaxial graphene with LEEM, LEED and XPS

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The graphitization of SiC(0001) surfaces via thermal silicon sublimation forms a carbon rich buffer layer (BL) at the interface between graphene and the substrate which is electronically insulating. This BL is a graphene-like layer which is covalently bound to the substrate [1]. Therefore it does not provide the electrical properties of freestanding graphene [2]. It has been shown that annealing the BL in an ultra-pure hydrogen atmosphere leads to a decoupling and conversion into quasi-freestanding monolayer graphene (QFMLG) [3]. Compared with regular epitaxial graphene on SiC the resulting QFMLG is characterized by an improved charge carrier mobility which makes it valuable for electronic applications [2].

A more precise investigation of short-term annealing of the BL in molecular hydrogen will increase the understanding of the initial stages of hydrogen intercalation and the decoupling of the BL.

This work reports on the investigation of stepwise decoupling of the BL by progressive intercalation using low energy electron microscopy (LEEM), low energy electron diffraction (LEED) and X-ray photoemission spectroscopy (XPS).

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## Growth and characterisation of graphene nanoribbons

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Graphene nanoribbons are one-dimensional allotropes of carbon. The reduced dimensionality leads to pronounced effects of quantum spatial confinement. Due to the relativistic-like dispersion of free carriers in graphene, the quantum confinement exhibits many well-distinguished phenomena in contrast to spatial quantization of parabolic bands. These are, for example, flat bands at the neutrality point in zigzag ribbons [1], or band gap opening and closing as a function of the ribbon width [2]. The reduced dimensionality also leads to reduced total amount of defects and impurities experienced by moving free carriers in perfectly hydrogen terminated ribbons. Such nearly ideal ribbons have been demonstrated by observing ballistic conductance [3]. Unfortunately, the real ribbons exhibit large edge roughness, and, due to the contamination from fabrication process, also additional scattering is induced by adsorbed e-beam/photo-resist residua.

We fabricate graphene ribbons by three methods. The conventional e-beam lithography is used as a reference technique. We improve this technique by protecting graphene by gold prior to e-beam resist coating. This approach reduces number of adsorbed residua on top of graphene ribbons. Next, we also etch SiC prior to growth of graphene ribbons to prepare so called sidewall graphene nanoribbons (SWGNR) [4,5]. The pre-patterned SiC annealed at high temperature preferentially sublimates at surfaces vicinal to (0001) face. The proper selection of growth time and temperature provides growth of one-dimensional graphene nanoribbons. The naturally quantum confined SWGNR are known to be terminated directly into SiC [6], thus providing nearly ideal ribbon termination. The protective gold layer also prevents adsorption of residual resist on top of graphene ribbons, thus eliminating carrier scattering.

The ribbons are characterized by atomic force microscopy (AFM), lateral force microscopy (LFM), electrostatic force microscopy (EFM), and scanning electron microscopy (SEM) and by Raman spectroscopy. We experimentally show how edge roughness of GNR can be reduced by properly setting the e-beam lithography, namely the dosage and number of e-beam exposures. A viability of new e-beam lithography techniques to obtain sub 20 nm GNR width is also discussed.

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# Quasi-free-standing graphene formed under high-pressure hydrogen

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Thermal decomposition of SiC (0001) is a technique to obtain wafer-scale graphene on the semi-insulating substrate with high quality. Epitaxial graphene obtained in this way has a buffer layer (BL) in the graphene-SiC interface and it degrades the electrical property of graphene. Hydrogen intercalation is a technique to decouple the BL from SiC substrate and convert it to quasi-free-standing graphene (QFSG) [1]. Although this is a promising approach to obtain QFSG, the conventional hydrogen treatment under atmospheric pressure is not enough to terminate all of the silicon dangling bonds. Here, we propose hydrogen intercalation under the high-pressure condition. Under high-pressure hydrogen gas, the intercalation reaction would proceed effectively, and would result in the carrier mobility improvement.

We firstly prepared BL samples by annealing the 4H-SiC (0001) substrate at 1500-1600 °C for 10 min under Ar 1 atm. Then, they were annealed again at 800 °C for 10 min under H<sub>2</sub> 1-3 atm for intercalation. Features of these samples were investigated by Raman spectroscopy, atomic force microscopy, and Hall-effect measurements.

Figure 1 shows Raman spectra of the samples before and after the hydrogen treatment. Before the hydrogen treatment, there were no graphene-derived features, and BL-derived broad peaks appeared around 1200-1700 cm<sup>-1</sup> [2]. After the hydrogen treatment, all samples showed 2D band around 2700 cm<sup>-1</sup>, which is the evidence of the conversion of BL to QFSG. The center position of 2D band reflects the strain in graphene [3]. Higher pressure condition resulted in lower Raman shift, which indicates that the compressive strain of QFSG obtained by higher hydrogen pressure was well-released. Presence of the D band indicates that graphene includes defects. These results also suggest that higher hydrogen pressure could produce higher quality QFSG.

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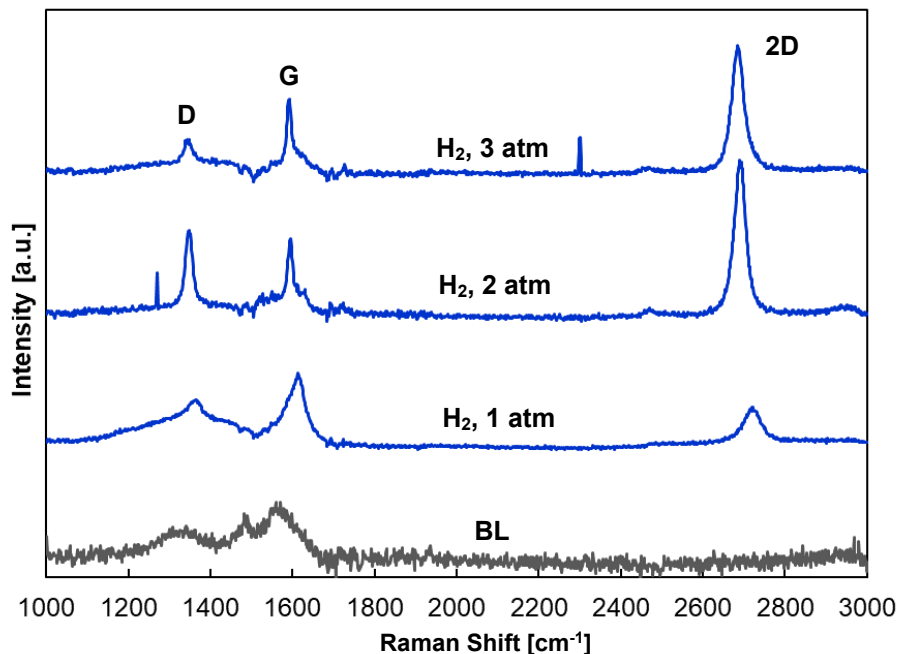


Figure 1 Raman spectra of BL and H-intercalated samples at different pressure conditions.

## Using XTEM to study the upper two nanometers of SiC(0001) under the graphene layers grown by silicon sublimation

Steffen Schulze, Markus Gruschwitz, Herbert Schletter and Christoph Tegenkamp

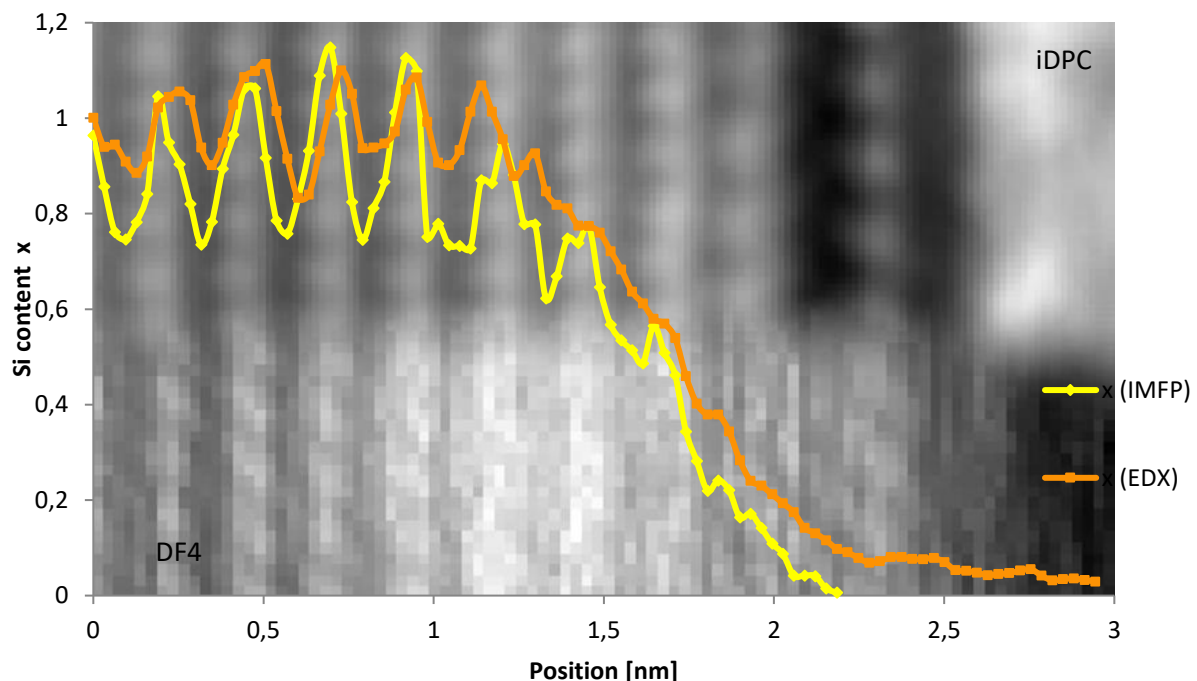
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Epitaxial graphene layers were fabricated by heating of 6H-SiC(0001) substrates. Due to significantly higher vapour pressure of the Si-fraction at 1800 K, a silicon depletion near to the surface develops culminating in sp<sup>2</sup>-hybridized graphene layers on top.

The cross section of the sub surface region has been imaged with differential phase contrasts (DPC) in the analytical Themis Z STEM showing the charge density distribution in the crystalline structure. Simultaneous EDX and EELS measurements revealed a defective crystal structure with silicon depletion to a depth of about one 6H-SiC unit cell below the surface.

For computing details of Si<sub>x</sub>C stoichiometry close to the surface, ZAF-corrected EDX net intensities have been used [1] as well as the inelastic mean free path (IMFP) calculated from EELS [2]. The figure below shows the silicon content in the sample surface region as derived from both methods in comparison with each other.

### Composition Si<sub>x</sub>C



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# Epitaxial graphene formation in N<sub>2</sub> ambient

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The formation of epitaxial graphene (EG) by Si sublimation method using a Si-faced SiC (0001) substrate is mainly performed in Ar ambient or in ultra-high vacuum [1]. According to previous researches, it is considered that the SiON is formed on the hydrogen-etched SiC surface in N<sub>2</sub> ambient [2], and EG formation on a Si-faced SiC substrate does not proceed. Therefore, it is expected that the EG can be formed even in an N<sub>2</sub> ambience by suppressing the formation of SiON in the Si sublimation process. In this research, a possibility of the EG formation in N<sub>2</sub> ambient using the suppression effect for the surface oxidation by the reduction materials such as AlN has been discussed.

The EG samples were formed in Ar ambient, or in N<sub>2</sub> ambient together with the AlN/SiC arranged around the SiC substrate. The Si sublimation process was performed at 1750 °C for 15 minutes by RF induction heating under the gas flow rate of 2.0SLM and the pressure of 400 torr.

Fig. 1 shows typical Raman spectra from the EG formed in (a) Ar ambient, (b) N<sub>2</sub> ambient together with AlN/SiC arranged around the SiC substrate, and (c) N<sub>2</sub> ambient, respectively. As shown in Fig. 1 (a), the G' band originating from the EG formed in Ar ambient was clearly observed around at 2700cm<sup>-1</sup>, but it could not be observed in the spectrum from the sample annealed in the N<sub>2</sub> ambient, as in Fig.1 (c). However, when the AlN/SiC is disposed upstream of the N<sub>2</sub> gas flow, a strong G' band from the EG could be observed, as shown in Fig. 1 (b). The results strongly indicate that the AlN thin film disposed upstream for the SiC substrate may suppress the SiON formation and forms the EG on the SiC substrate.

In conclusions, we have investing the possibility of the EG formation in N<sub>2</sub> ambient by suppressing surface oxidation using reduction material such as AlN and we have successfully formed the EG even in N<sub>2</sub> ambient.

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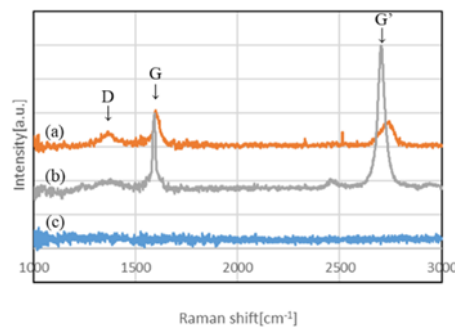


Fig. 1 Typical Raman spectra from EG samples formed in (a) Ar ambient, (b) N<sub>2</sub> ambient with AlN/SiC and (c) N<sub>2</sub> ambient only, respectively.

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## Optical spectroscopy of graphene nanoribbons

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Graphene nanoribbons are promising candidates for transport of spin currents on long distances. Hence using them as interconnects in spintronics can be further step towards a real world application of graphene [1].

We present a far infrared spectroscopic (FIR) study of graphene nanoribbons fabricated by three distinct methods. First, conventional ebeam lithography [2] is used. This method is known to produce rough edges, hence deteriorating transport properties of free carriers. Second, we study ribbons prepared by ebeam lithography using gold protective mask [3]. This method allows reducing contamination of ribbons by ebeam resist. Third, we study so called sidewall ribbons [4] where both disadvantage of rough edges and resist contamination is treated by first prepaterning SiC and by natural attachment of the ribbon to the underlying substrate.

The main goal of this FIR study is to exploit the large oscillator strengths at room temperature of the plasmon resonances [5] of the graphene nanoribbons in order to characterize their properties such width and overall quality among three above mentioned kinds of fabrication. The results of FIR study are compared with micro Raman mapping [6] and atomic force microscopy.

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# Graphene nanoribbons on macro-facets of vicinal 6H-SiC(0001)

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Graphene is considered for the switching devices applications, which require a band gap in electronic structure. It is possible to open band gap in narrow graphene nanoribbons (GNRs) [1]. The GNRs produced by top-down methods reveal fatal limitations in terms of width control and density of defect. Thus, bottom-up approaches are demanded to solve these problems. Palacio *et al.* proposed the method to tailor GNRs on the SiC (1-10n) trench sidewalls produced by lithography [2]. Here, we propose a method for producing GNRs on self-organized SiC macro-facets by surface decomposition of vicinal SiC (0001) substrate.

6H-SiC(0001) substrates with 15° off-axis angle toward [1-100] were used. High temperature H<sub>2</sub> gas etching was firstly performed to produce typical nanofacet/terrace surface as often observed on vicinal 6H-SiC(0001) surfaces [3]. The substrates were then post-annealed in Ar gas atmosphere at 1500 °C for various durations resulting in macro faceting and graphene formation. Figure 1 is a STM-height image on a macro-facet with single graphitic layer. Two different kinds of structure appear, one being smooth and the other having bumpy features with ~1:1 areal ratio. These are predicted to be a free-standing graphene (GNR) and a kind of buffer layer, respectively. Figure 2 is the LEED pattern taken with the incident electron beam normal to macro-facet (~27°). Super-spots of SiC, graphene, and buffer layer due to facet periodic structure of 3.4 nm are visible, suggesting GNRs and buffer layer ribbons are alternately appeared. Figure 3 shows Raman spectrum of macro-facet area. It indicates D (1380 cm<sup>-1</sup>), G-bands (1610 cm<sup>-1</sup>), and relatively weak peaks due to the buffer layer. Additionally, it is noted 2D peak is absent, characteristic of narrow GNRs [4]. We will show further analyses on the GNRs using polarized Raman and angle-resolved photoemission spectroscopy (ARPES).

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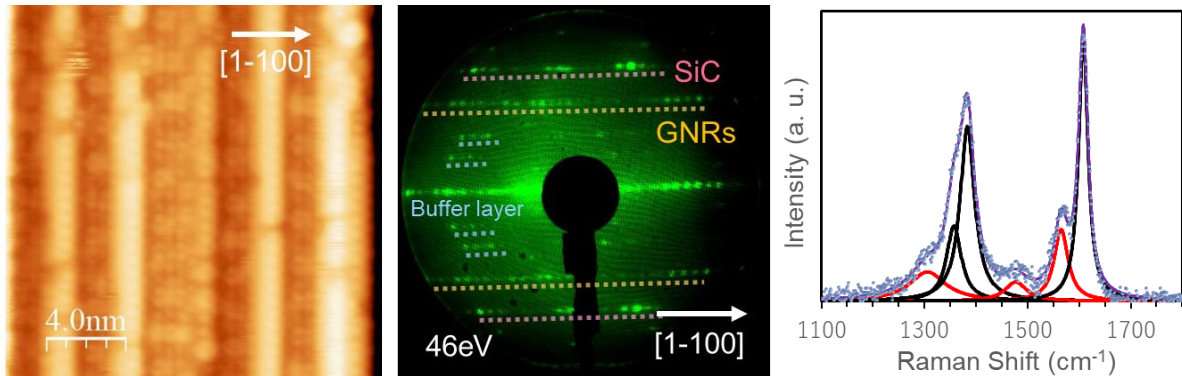


Fig. 1: STM height image of facet region.

Fig. 2: A LEED pattern at the incident electron beam normal to the facet. Dotted lines indicate the super-spots due to periodic features on the facet as are observed in Fig. 1.

Fig. 3: Raman spectrum of the facets. Black and red lines by peak separation are originated from GNRs and the buffer layer, respectively.

## Epitaxial Growth of High Quality Armchair Graphene Nanoribbons

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Graphene nanoribbons (GNRs) are considered to be the fundamental building blocks for future carbon-based nanoelectronics. The functionality of GNRs is governed by the detailed atomic structure of their edges. Namely, GNRs with zigzag edges demonstrate 1D single-channel ballistic transport at room temperature [1], while those with armchair edges promise a width-dependent semiconducting behavior [2]. In this work, we present a method for scalable template growth of high quality armchair GNRs on the sidewalls of predetermined SiC mesa structures. The quality of the grown ribbons is confirmed by comprehensive characterization techniques such as AFM, LEED, and ARPES. The growth process results in SiC mesa walls decomposing into unit-cell high microsteps that host a number of well-aligned 2-3 nm wide armchair GNRs. We observe the presence of different sub-bands in their electronic band structure with a Fermi surface consisting strictly of a straight line, which is characteristic for 1D confined systems. Our findings provide a solid ground for further theoretical assessment and a deeper understanding of quantum confinement phenomena.

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# Quadratic to linear magnetoresistance crossover in epitaxial graphene on SiC

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Resistance is one of the most experimentally studied properties of solid state materials. In spite of its wide spread usage, the resistance in transverse magnetic field very often shows unexpected behavior. The major mystery follows from prediction of the simplest Drude model. Inverting conductivity tensor leads to field independent longitudinal resistivity and linear in field off-diagonal resistivity. The classical Hall Effect stands behind the field dependence of the off-diagonal resistivity. However, the expected field independent diagonal, or longitudinal, resistivity is observed very rarely [1-7]. The common explanation for so called positive quadratic resistivity is given in terms of finite size geometry of the sample. The linear magnetoresistance, however, remains elusive [1-3]. The most common physical model to explain the linear magnetoresistance is to consider the so called resistance network model [1,3,4,5]. The weak point of the resistance network model is the requirement on large variance of charge density. The charge density fluctuation on the order of twice the charge density requires potential energy fluctuations on the order of twice the Fermi level. Such large Fermi level variations are ten times above the experimentally determined values of surface potential fluctuations in epitaxial graphene, and, it is entirely inapplicable for ultra-high mobility electron gas [6] or metals. It would also require ambipolar conductivity, unfavoured by typically linear-in-field Hall resistance. Instead, we show, that the finite sample size is behind the linear magnetoresistance. The crossover from quadratic to linear magnetoresistance is given by the low-field to high-field crossover. The analytical argumentation by Lorentz force is given, and, we support our conclusion by numerical Finite Element simulation of the current distribution in magnetic field by solving the continuity equation. We also show that the current streamlines constriction is not given by particular boundary conditions; however, it is an immediate consequence of the junction between materials with two different conductivities and/or mobilities. The finite sample size effect on a non-saturating linear magnetoresistance is experimentally demonstrated in epitaxial graphene on SiC(0001). The sharp low-field to high-field crossover fits well with the inverse value of carrier mobility. The low-field quadratic magnetoresistance is described by square of the mobility, and, the high-field linear magnetoresistance scales linearly with mobility.

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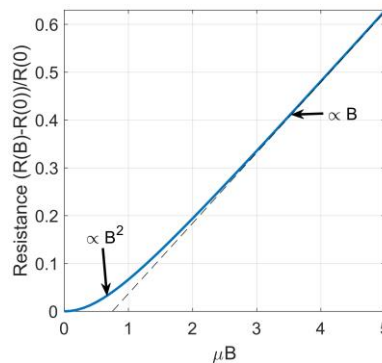


Fig. 1: Numerical simulation showing the quadratic to linear magnetoresistance crossover.

# Effect of hydrogen intercalation on graphene mechanical properties

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We have studied the effect of hydrogen intercalation on the mechanical properties of the buffer layer, single layer graphene (SLG), quasi-free standing monolayer graphene (QFSMLG) and quasi-free standing bilayer graphene (QFSBLG). The buffer layer contains 30% of  $sp^3$  bonded carbon, which is bonded to the Si in SiC. This interface pins the Fermi level in the graphene layer. Also, this layer reduces the carrier mobility due to the interaction between carrier in graphene and phonons in SiC. Hydrogen intercalation saturates the Si-C bonds between SiC and buffer layer. It causes the change of  $sp^3$  bonds back into  $sp^2$  bonds, and this results in QFSMLG and QFSBLG which is formed from buffer layer and SLG, respectively.

The buffer layer, SLG, QFSMLG and QFSBLG samples were grown on the Si-face of on-axis SiC substrate using thermal decomposition method. The QFSMLG and QFSBLG samples were formed from the buffer layer and SLG after hydrogen intercalation [1,2].

The micro-Raman spectroscopy was used to determine the quality of the samples.

Friction force microscopy [3], Kelvin probe force microscopy (KPFM) [4] and force versus indentation depth [5] measurements were performed to explore the samples' properties. During friction measurements the relative humidity was about 15%, and temperature was kept at 24°C. The samples were measured under seven normal loads between -10 and 50 nN with steps of 10 nN. The dimension of the scanned areas were 1,5x1,5  $\mu m^2$ , and the scan speed was 6  $\mu m$  per second. The topography and friction map of the buffer layer are shown in Fig. 1. Fig. 2 displays the evolution of friction force as a function of normal load, for the 4 samples. This graph shows that the hydrogen intercalation leads to lower friction, which corresponds to the change of  $sp^3$  bonds back into  $sp^2$ .

It was recently discovered that the SLG transforms into a diamond-like ultra-stiff and ultra-hard phase under pressure [6]. To understand the influence of the buffer layer in this phenomenon, we measured the stiffness of the samples with and without buffer layer using the AFM-based Modulated Angstrom indentation technique and a diamond coated silicon tip. KPFM was used as a method to estimate the influence of intercalation on the work function of samples, which is related to the position of the Fermi level.

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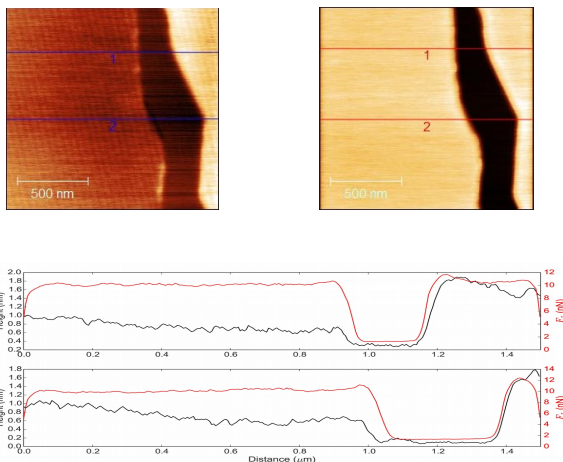


Fig. 1: Topography map (upper left) and friction map (upper right) of the buffer layer. The bottom graph shows the profiles from topography and friction maps.

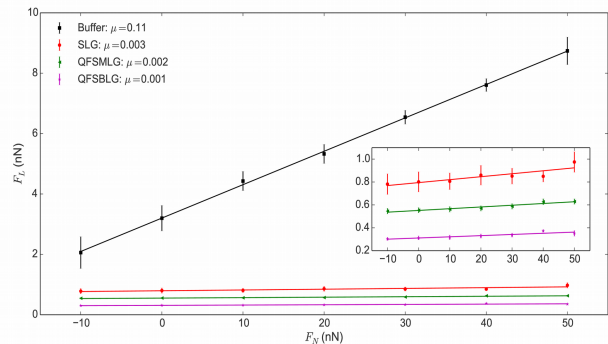


Fig. 2: Friction force as a function of normal load.

# Electronic states of graphene transferred onto the buffer layer on SiC

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Graphene can be grown epitaxially on the semi-insulating substrate in large scale using thermal decomposition of SiC. Therefore, it suits applications for high-speed transistors. However, the carrier mobility in epitaxial graphene (EG) on SiC at room temperature is reduced compared to that at low temperatures due to the carrier scattering by phonons in the buffer layer (BL) [1]. In order to improve the carrier mobility of EG, we investigated the effects of the interface and the type of graphene on the electronic states of graphene on SiC. For this purpose, in this study, we transferred EG and CVD graphene (CVDG) onto different substrates [2]. The electronic band structures and the electrical properties of these samples by angle resolved photoemission spectroscopy (ARPES) and Hall-effect measurements.

We prepared EG by heating the 4H-SiC (0001) substrate in Ar at 1720 °C, which was homogeneous over an area of  $5 \times 5$  mm<sup>2</sup>. As the first step of transfer, EG was coated with a layer of Au (about 200 nm thick). Then, the Au/EG film was exfoliated from the BL/SiC substrate using the thermal release tape. This EG/Au/tape film was put on a target substrate (SiC wafer with BL, or silicon wafer with 90nm-thick thermal oxide), and we removed the tape by heating. Finally, Au thin film was etched away using I<sub>2</sub>/KI solution. CVDG was grown on Cu(111)/sapphire and transferred on to the same substrates. We investigated the features of the samples using atomic force microscopy (AFM), Raman spectroscopy, Hall-effect measurements, and ARPES measurements. The ARPES measurements were done after annealing for four hours at about 573 K under ultra-high vacuum.

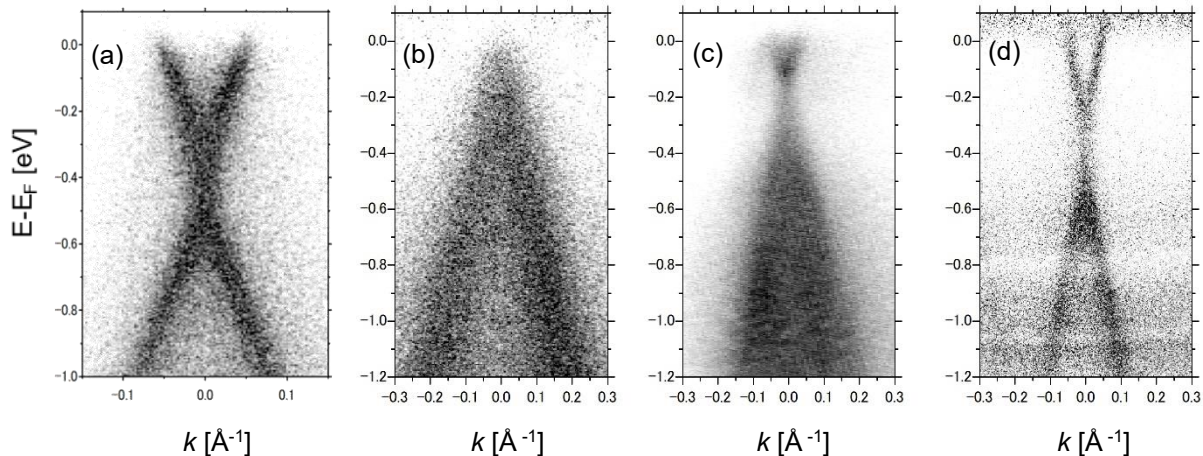
In the Raman spectra of the substrates before the transfer process, broad peaks were found at 1200 ~ 1600 cm<sup>-1</sup>, and 2D band was not found. This indicates that BL covers the surface of these substrates. After the transfer process, we found sharp G and 2D band at about 1595 and 1680 cm<sup>-1</sup>, respectively. Figures 1(a), (b), (c) and (d) show ARPES spectra of EG, exfoliated EG (e-EG) on Au, transferred EG (t-EG) on BL, and t-CVDG on BL, respectively. Clear Dirac cone was observed in each sample. In e-EG, the Dirac point was located at about 0.07 eV below the Fermi energy, which indicates low electron concentration compared to EG. The Dirac point in t-EG on BL and t-CVDG on BL were at about -0.20 and -0.34 eV, respectively. These results indicated that graphene on BL was electron-doped, regardless of the graphene growth techniques. On the other hand, our Hall-effect measurements revealed that the dominant carriers in t-EG and t-CVD were hole. This hole-doping was probably attributed to adsorbents such as H<sub>2</sub>O or O<sub>2</sub>, which were adsorbed in air after ARPES measurements.

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Figs. 1: ARPES spectra around the K point of (a) EG [3], (b) e-EG on Au, (c) t-EG on BL, (d) t-CVDG on BL.

# Phonon dispersion of bulk MoS<sub>2</sub> determined by high resolution electron energy loss spectroscopy

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With the unbroken interest in graphene there is also increasing scientific attention for related two-dimensional materials such as transition metal dichalcogenides (TMDCs). Combination of two-dimensional materials like these are predicted to lead to novel possible applications and device structures. Basic properties like, e.g. electronic structure as well as phonon and plasmon dispersion, are of fundamental interest in this respect. High resolution electron energy loss spectroscopy (HREELS) is a powerful tool for the investigation of the phononic structure of a solid. Here we use HREELS to study the phonon dispersion of molybdenum disulfide (MoS<sub>2</sub>).

A clean surface of a MoS<sub>2</sub> bulk crystal was prepared by mechanical cleavage and mild annealing in ultra-high vacuum (UHV). Using low-energy electron diffraction (LEED), the sample was aligned in the high symmetry directions  $\Gamma$ -K and  $\Gamma$ -M of the 2D Brillouin zone, respectively, and subsequent HREELS measurements were performed in off-specular geometry with a fixed incident electron energy and angle by varying the detector angle of the spectrometer. The spectra were fitted using a homemade routine and matching Voigt-profiles to the HREELS spectra for each resolvable phonon mode. The results are in good agreement with recent DFT-calculations from literature [1] (see Fig. 1) but certain differences in comparison to an earlier HREELS study [2] were noted.

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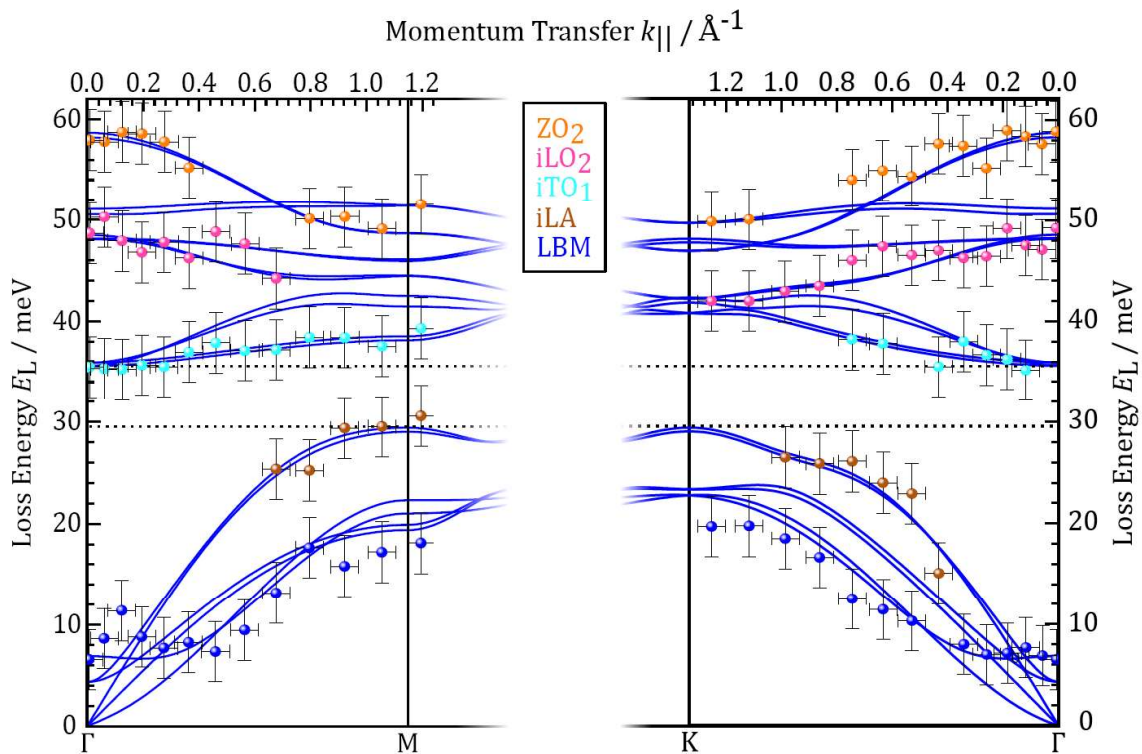


Fig. 1: Comparison of phonon modes measured with HREELS and recent DFT-calculations [1].

## Periodically rippled graphene formed on 4H-SiC m-plane surface

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Takushi Iimori<sup>2</sup>, Fumio Komori<sup>2</sup>, Satoru Tanaka<sup>1</sup>

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<sup>2</sup> The Institute for Solid State Physics, the University of Tokyo, Chiba, Japan

The formation of periodically aligned graphene nanoribbons<sup>[1]</sup> by utilizing surface nanostructure uniquely appeared on vicinal SiC(0001)<sup>[2]</sup> is promising for switching device applications, which require a band gap. Periodic ripples in graphene are also effective to form a band gap due to periodic potential modulation induced by a pseudo magnetic field<sup>[3]</sup>. Clean SiC m-plane surface reveals periodic corrugation in nanoscale. In the present study, we utilize this feature to form rippled graphene. We have found that monolayer graphene transferred on the m-plane surface may exhibit periodically rippled structure and we expect corresponding modification of electronic structure.

Initially, a 4H-SiC m-plane substrate was etched by H<sub>2</sub> gas and was examined by atomic force microscope (AFM) and scanning tunneling microscope (STM). The surface have shown periodic corrugation of 1 nm period, corresponding to one unit cell length along c-axis of 4H-SiC, as seen in the STM image of Fig. 1(a). The surface atomic model is depicted in Fig. 1(b), which is similar to a model proposed by Seyller *et al.*<sup>[4]</sup> Monolayer graphene was separately grown on 4H-SiC(0001) by CVD using C<sub>2</sub>H<sub>4</sub>/Ar gas and was exfoliated from the substrate using sputtered Cu thin film and adhesive tape. Then, the Cu/graphene film was transferred onto the 4H-SiC m-plane surface, followed by the removal of the Cu film by acid solution. The sample was first examined by low-energy electron diffraction (LEED), however, it showed blurred graphene diffraction spots and high background intensity. After annealing at 1000 °C in UHV, clear diffraction spots from graphene appeared as shown in Fig. 2. Notice here that satellite diffraction spots are also visible (see arrows). These are superlattice spots due to 1 nm periodicity of corrugated graphene, as a result of superior adhesion between graphene and SiC surface after the annealing process in UHV. We will show the results of micro-Raman spectroscopy and angle-resolved photoemission spectroscopy (ARPES) to reveal new features of electronic structure.

### References:

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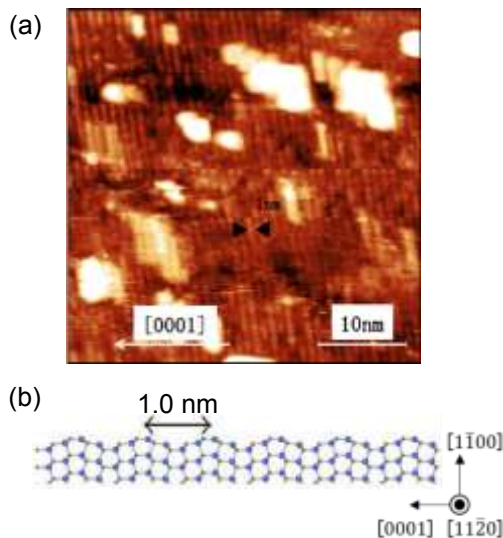


Fig. 1: (a) STM image of 4H-SiC m-plane surface showing periodically (1 nm) corrugated structure. (b) Cross section of atomic structure model of SiC m-plane surface.

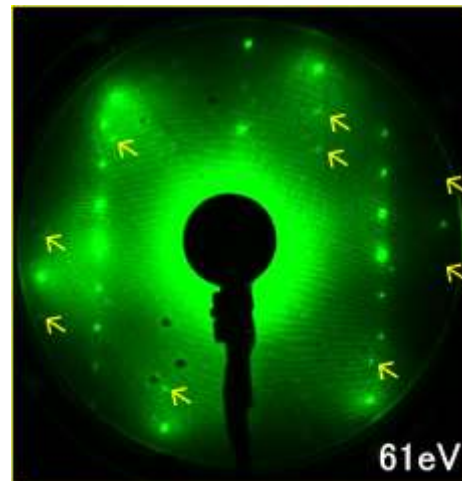


Fig. 2: LEED pattern of graphene transferred onto SiC m-plane surface. Arrows indicate superlattice reflections due to 1 nm periodicity of corrugated graphene.







## Schedule

	Sunday, 25.08.2019	Monday, 26.08.2019	Tuesday, 27.08.2019	Wednesday, 28.08.2019
8.45 - 9.00		Opening remarks		
9.00 - 9.45		Jobst	Ding	Pierz
9.45 - 10.15		Sinterhauf	Schädlich	Lin
10.15 - 10.45		Break	Break	Break
10.45 - 11.15		Heilmann	Zakharov	Imamura
11.15 - 11.45		Power	Göhler	Karakachian
11.45 - 12.30		Seneor	Gemming	Tzalenchuk
12.30 - 13.30		Lunch	Lunch	Closing Remarks Lunch
13.30 - 14.15		Iacopi	de Heer	
14.15 - 14.45		Vagin	Gerstmann	14.00 Excursion
14.45 - 15.15		Nhung Nguyen	Wells	
15.15 - 15.45		Break	Break	
15.45 - 16.15		Pradeepkumar	Tanaka	
16.15 - 17.00		Wenderoth	Norimatsu	
17.00 -	16:00 Registration and Get-Together	Poster	Poster	Conference Dinner
		Aprojanz, Feijo, Gruschwitz, Krone, Kunc, Sakakibara, Schulze, Takeuchi	Dedic, Fukuma, Karakachian, Kunc, Reihon, Sato, Schwarz, Uotani	